

US EPA RECORDS CENTER REGION 5



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INITIAL SAMPLING PLAN
FOR
REILLY TAR & CHEMICAL CORP.
N.P.L. SITE
ST. LOUIS PARK, MINNESOTA

SUBMITTED OCTOBER, 1986

AMENDED FEBRUARY, 1987

AMENDED MARCH, 1988

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**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604**

REPLY TO THE ATTENTION OF:

MEMORANDUM

5SMQA-10

DATE: May 12, 1988

SUBJECT: Approval of Quality Assurance Project Plan Groundwater
and GAC Plant for the Reilly Tar and Chemical Corporation
Superfund Site, St. Louis Park City, Minnesota

FROM: Andrea Jirka, Chief *[Signature]*
Monitoring and Quality Assurance Branch

TO: Norman Niedergang, Chief
CERCLA Enforcement Section

Attention: Erin Moran, RPM

This memorandum transmits our QAS office's approval of the Quality Assurance Project Plan (QAPP) for groundwater and GAC plant monitoring at the Reilly Tar and Chemical Corporation Superfund site located in St. Louis Park City, Minnesota. This approval is provided after QAS office has received the changed pages required to facilitate this subject QAPP for approval on May 11, 1988. Please note that we also have made a minor change, prior to this approval, on page 8 of 68, Section 4. A copy of this changed page is attached for your use. Please incorporate it in the QAPP.

The original signature page is included. Please have the Remedial Project Manager provide final sign off.

Attachment

cc: K. Chiu, TSU
C-W. Tsai, QAS

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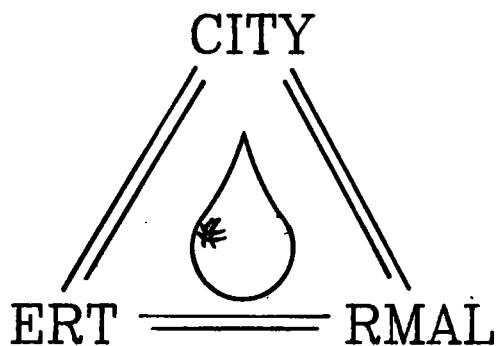
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QUALITY ASSURANCE UNIT

APR 05 1988

ENVIRONMENT SERVICES DIVISION



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QUALITY ASSURANCE BRANCH

APR 05 1988

ENVIRONMENT SERVICES DIVISION

SECTION A

SITE MANAGEMENT PLAN

INTRODUCTION

Ground water in the City of St. Louis Park, Minnesota has been contaminated by activities at a coal-tar distillation and wood preserving plant operated from 1917 to 1972. Numerous previous studies have identified polynuclear aromatic hydrocarbons (PAH) present in various aquifers beneath St. Louis Park and adjacent communities.

The United States Environmental Protection Agency (EPA), the Minnesota Pollution Control Authority (MPCA), the Minnesota Department of Health (MDH), the City of St. Louis Park (SLP), and Reilly Tar & Chemical Corporation (Reilly) have agreed to acceptable water quality criteria for PAH. These criteria, as incorporated into the Consent Decree, include the following concentration levels:

	<u>Advisory Level</u>	<u>Drinking Water Criteria</u>
o Sum of benzo(a) pyrene and dibenz(a,h) anthracene	3.0 ng/l*	5.6 ng/l
o Carcinogenic PAH	15 ng/l	28 ng/l
o Other PAH	175 ng/l	280 ng/l

*or the lowest concentration that can be quantified, whichever is greater

In conjunction with the implementation of remedial measures to limit the spread of contaminants, a granular activated carbon (GAC) treatment system has been installed to treat water from St. Louis Park (SLP) wells 10 and 15. Further provisions of the Remedial Action Plan (RAP) call for long-term monitoring of the influent and effluent of the GAC treatment plant and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH and/or phenolics in the ground water. The analytical data will be used to evaluate contamination by comparing the levels of PAH and/or phenolics found in the various samples with historical water quality data and with water quality criteria established in the Consent Decree-RAP. The specific objectives of the sampling and analysis program, and therefore, the intended end use of the data vary slightly for the different aquifers being monitored in accordance with the Consent Decree-RAP.

The GAC plant monitoring is being done to assess and continuously evaluate the performance of the treatment system. Analytical results for influent and effluent samples will be compared to the drinking water criteria for PAH as established in the Consent Decree-RAP. Based on these

comparisons, decisions will be made on: 1) possible modifications to the treatment system (e.g., adding another carbon column), 2) system operations (e.g., when the carbon should be replaced), and 3) cessation of the treatment system, if desired, when sufficiently low concentrations of PAH in influent samples are demonstrated.

The objective of sampling the four existing Mt. Simon-Hinckley Aquifer municipal drinking water wells and any new Mt. Simon-Hinckley Aquifer municipal drinking water wells installed within one mile of well W23, and analyzing for PAH, is to assure the continued protection of these wells from PAH resulting from activities of Reilly at the site. The analytical data will be used to make comparisons between the levels of PAH found in the Mt. Simon-Hinckley Aquifer, and the drinking water criteria established in the Consent Decree-RAP.

The objective of sampling and analyzing the Ironton-Galesville Aquifer source control well (W105) is to assess the levels of PAH in the discharge from W105 when it is pumping a monthly average of 25 gallons per minute. The data will be used to compare the concentration of total PAH in the samples to a cessation criterion of 10 micrograms per liter of total PAH established in the Consent Decree-RAP. Also, if any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23, then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the well from PAH resulting from the activities of Reilly at the site. The analytical data would be used to compare the levels of PAH found in potential Ironton-Galesville Aquifer drinking water wells to the drinking water criteria established in the Consent Decree-RAP.

The objectives of monitoring the many Prairie du Chien-Jordan Aquifer wells, including municipal drinking water wells, private or industrial wells, and monitoring wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control systems, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the site. The analytical data will be used to compare the levels of PAH in the Prairie du Chien-Jordan aquifer to historical PAH data and to various criteria established in the Consent Decree-RAP (e.g., drinking water criteria for drinking water wells, and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23). Analytical data for samples of the discharge from gradient control well SLP4 will be compared to discharge limitations in an NPDES permit that will be applied for at the conclusion of a Feasibility Study to determine the appropriate disposition of SLP4 discharge. Water level data will be used to evaluate ground-water flow patterns in the Prairie du Chien-Jordan Aquifer.

The objective of monitoring St. Peter Aquifer wells is to determine the nature and extent of PAH in the St. Peter Aquifer resulting from the activities of Reilly at the site. The analytical data will

be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data and to the drinking water criteria established in the Consent Decree-RAP. Water level data will be used to evaluate ground-water flow patterns in the St. Peter Aquifer.

The objectives of monitoring the Drift-Platteville Aquifer wells are to: 1) monitor the distribution of PAH and phenolics in the aquifer, thus evaluating the source and gradient control systems, and 2) to further define the nature and extent of PAH and phenolics in the Northern Area of the Drift-Platteville Aquifer resulting from the activities of Reilly at the site. The analytical data will be used to compare levels of PAH and phenolics in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the Consent Decree-RAP for PAH and phenolics. Water level data will be used to evaluate ground-water flow patterns in the Drift-Platteville Aquifer.

This Site Management Plan outlines the scope of work to be performed in order to monitor the ground water in the St. Louis Park, MN area in accordance with the Consent Decree - RAP related to the Reilly Tar & Chemical Corp. N.P.L. site. Included in this plan are: 1) the identity of wells to be monitored, 2) the schedule for ground-water monitoring, and 3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting.

The time period covered by the Initial Sampling Plan is from the date of its acceptance and approval by the agencies, to December 31, 1988. The first subsequent Sampling Plan (RAP section 3.3) will be submitted by October 31, 1988, covering the 1989 calendar year.

This Plan incorporates the requirements of RAP Sections 3.2, 3.3, 4.3, 5.1, 6.1.4, 7.3, 8.1.3, 9.1.3, 9.2.3, 9.3.3, and 9.6. Some of the sampling required under RAP Section 4.3 (Monitoring the GAC System) has already taken place prior to the Effective Date. Therefore, only the monitoring that will take place from the approval date of this Initial Sampling Plan through December 31, 1988 is included in this Plan.

SAMPLING SCHEDULE

The actual dates of ground-water monitoring are based on the timing of activities conducted under the RAP, and these dates cannot be predicted now with certainty. For example, except for the interim monitoring of the GAC plant, no monitoring will take place until this plan is approved. Therefore, the proposed sampling schedule outlined in this sampling plan indicates the starting criteria and the frequencies of sampling as outlined in the RAP to determine when the wells are sampled (Tables 1 and 2). In general, the sampling schedule will be constructed to allow economies of scale in the field and in the laboratory by grouping the various monitoring events described by the RAP as much as possible. Samples will be collected within the time periods indicated on Tables 1 and 2, and all parties will be given two weeks notice in advance of routine sampling.

Tables 1 and 2 summarize the ground-water monitoring schedule for the period through December 1988, and represent the minimum monitoring program that is likely to occur during the year. However, additional sampling will take place if treated water from the GAC plant or ground-water from active municipal drinking water wells exceeds the drinking water criteria established in the Consent Decree-RAP. This additional sampling is described in Sections 4 and 12 of the RAP, and are reproduced in Appendix A of this Site Management Plan.

The duration of field sampling events will depend on the number and type of wells to be sampled. For estimating purposes, it is assumed that between 10 and 20 active pumping wells (e.g., municipal, industrial, or gradient/source control wells), or between 4 and 8 monitoring wells can be sampled in one day. It is a reasonable expectation that most sampling events will take place over the better part of a week, and some sampling may be done over a longer time frame.

**TABLE 1. INITIAL SAMPLING PLAN GAC PLANT
MONITORING SCHEDULE ^(a)**

<u>RAP Section</u>	<u>Sampling Points</u>	<u>Start of Monitoring</u>	<u>Sampling Frequency</u>	<u>Analyses</u>^(b)
4.3.1 (C)	Treated water(TRTD)	Date of plan approval	Monthly	PAH(ppt) ^(c)
4.3.3 (C)	Feed water(FEED)	Date of plan approval	Quarterly	PAH(ppt)
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH(ppt)
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds in EPA Test Method 625.

(a) This schedule does not include certain contingencies (eg. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1988. Sections 4 and 12 of the RAP outline the additional sampling that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. The location of the GAC plant is shown in Figure 1.

(b) Lists of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds in EPA Test Method 625 are provided in the QAPP. Field blanks will be collected and analyzed at a frequency of one per day. Duplicate samples will be collected and analyzed at a frequency of one per 10 samples.

(c) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

**TABLE 2. INITIAL SAMPLING PLAN GROUND-
WATER MONITORING SCHEDULE ^(a)**

<u>Source of Water</u>	<u>RAP Section</u>	<u>Sampling^(l) Points</u>	<u>Start of Monitoring</u>	<u>Sampling Frequency</u>	<u>Analyses^(b)</u>
Mt. Simon-Hinckley Aquifer	5.1	SLP11, SLP12, SLP13, SLP17	Within six months of Effective date ^(g)	Annually	PAH (ppt) ^(c)
	5.3.2	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Ironton-Galesville Aquifer	6.1.4	W105 W38 ^(e)	Start of pumping	Quarterly	PAH (ppb) ^(d)
	6.2.1	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Prairie du Chien-Jordan Aquifer	7.3 (A)	SLP4	Start of pumping	Quarterly	PAH (ppt) ^(h) phenolics
	7.3 (B)	W23	Start of pumping	Quarterly	PAH (ppb)
	7.3 (C)	SLP6, SLP7 or SLP9, W48	Date of plan approval	Quarterly	PAH (ppt)
	7.3 (D) ^(m)	AHM or MGC ⁽ⁱ⁾ , E2, E13, H3, SLP10 or SLP15, SLP14, SLP16, W402 ^(j) W403, W119	Date of plan approval	Semi-annually	PAH (ppt)
	7.3 (E) ^(m)	SLP5, H6, E3, E15, MTK6, W29, W40, W70, W401 ^(j)	Date of plan approval	Annually	PAH (ppt)
	7.3 (F)	W112, W32, SLP8, SLP10, E4, E7	Date of plan approval	Quarterly	No chemical analyses ^(f)
St. Peter Aquifer	8.1.3	SLP3, W14, W24, W33, W122, W129 W133, P116, plus 5 new wells	Within 30 days of installing new wells	Once	PAH (ppt)
		SLP3 plus six of the wells listed above ⁽ⁿ⁾	Within 6 months of above	Once	PAH (ppt)

TABLE 2 (continued)

<u>Source of Water</u>	<u>RAP Section</u>	<u>Sampling^(l) Points</u>	<u>Start of Monitoring</u>	<u>Monitoring Frequency</u>	<u>Analyses^(b)</u>
Drift-Platteville Aquifer	9.1.3 and 9.2.3	Source and gradient control wells (3 wells)	Start of pumping	Quarterly	PAH (ppb) and total phenols
	9.3.3	W131, W136, plus 6 new wells	Within 30 days of well installations	Once	PAH (ppb) and total phenols
	9.3.3	W131, W136 plus 6 new wells	Within 6 months of above	Once	PAH(ppb) and total phenols
	9.6	Drift: W2, W6, W10, W11, W12, W116, W117, W128, W135, W136, P109, P112, Platteville: W18, W1, W19, W20, W27, W101, W120, W121, W124, W130, W131, W143, plus 6 new wells	Concurrent ^(k) with 9.3.3 sampling	Concurrent ^(k) with 9.3.3 sampling	PAH (ppb) and total phenols

- (a) This schedule does not include certain contingencies (eg. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1988. Section 12 of the RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. Field blanks will be collected at a frequency of one per day, and one duplicate sample will be collected for every 10 samples.
- (b) Lists of parameters and descriptions of the methods for analysis of PAH, phenolics, and expanded analyses are provided in the QAPP. Water levels will be measured each time samples are collected for analysis, except for those wells which prove to be inaccessible for such measurements.
- (c) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.
- (d) ppb = parts per billion. This signifies analysis by EPA Method 625. If analytical results for individual wells are below 20 micrograms per liter (20 ppb) using this method, then the part per trillion method will be used on subsequent monitoring rounds.
- (e) Water levels in W38 will be measured each time W105 is sampled.
- (f) Water levels only (no monitoring) will be measured at these wells, except for those wells which prove to be inaccessible for such measurements.

TABLE 2 (continued)

- (g) Or within 30 days of the approval date of this Plan, whichever is later.**
- (h) SLP4 analytical program will be determined by the results of the Feasibility Study.**
- (i) AHM = American Hardware Mutual, MGC = Minikahda Golf Course.**
- (j) Wells W401, W402, and W403 may or may not be available for sampling at the same time as the other wells on these lists. They will be sampled in conjunction with the monitoring performed in accordance with the schedule shown, once they are available for sampling.**
- (k) If any of the wells listed here become damaged, destroyed, or otherwise unsuitable for sampling, alternate wells will be selected by the Project Leaders for monitoring.**
- (l) Sampling points are located on the maps shown in Figures 1 through 5. Letter prefixes to well codes are defined as follows:**

 - W - 4-inch monitoring well**
 - P - monitoring piezometer**
 - PB - 2-inch monitoring well**
 - SLP - St. Louis Park supply well**
 - E - Edina supply well**
 - H - Hopkins supply well**
 - MTK - Minnetonka supply well**
- (m) Water level measurements will be made quarterly at these wells, except for those wells which prove to be inaccessible for such measurements.**
- (n) The six St. Peter Aquifer monitoring wells that will be monitored according to RAP Section 8.1.3 will be selected by the Project Leaders based on the results of the first monitoring round.**

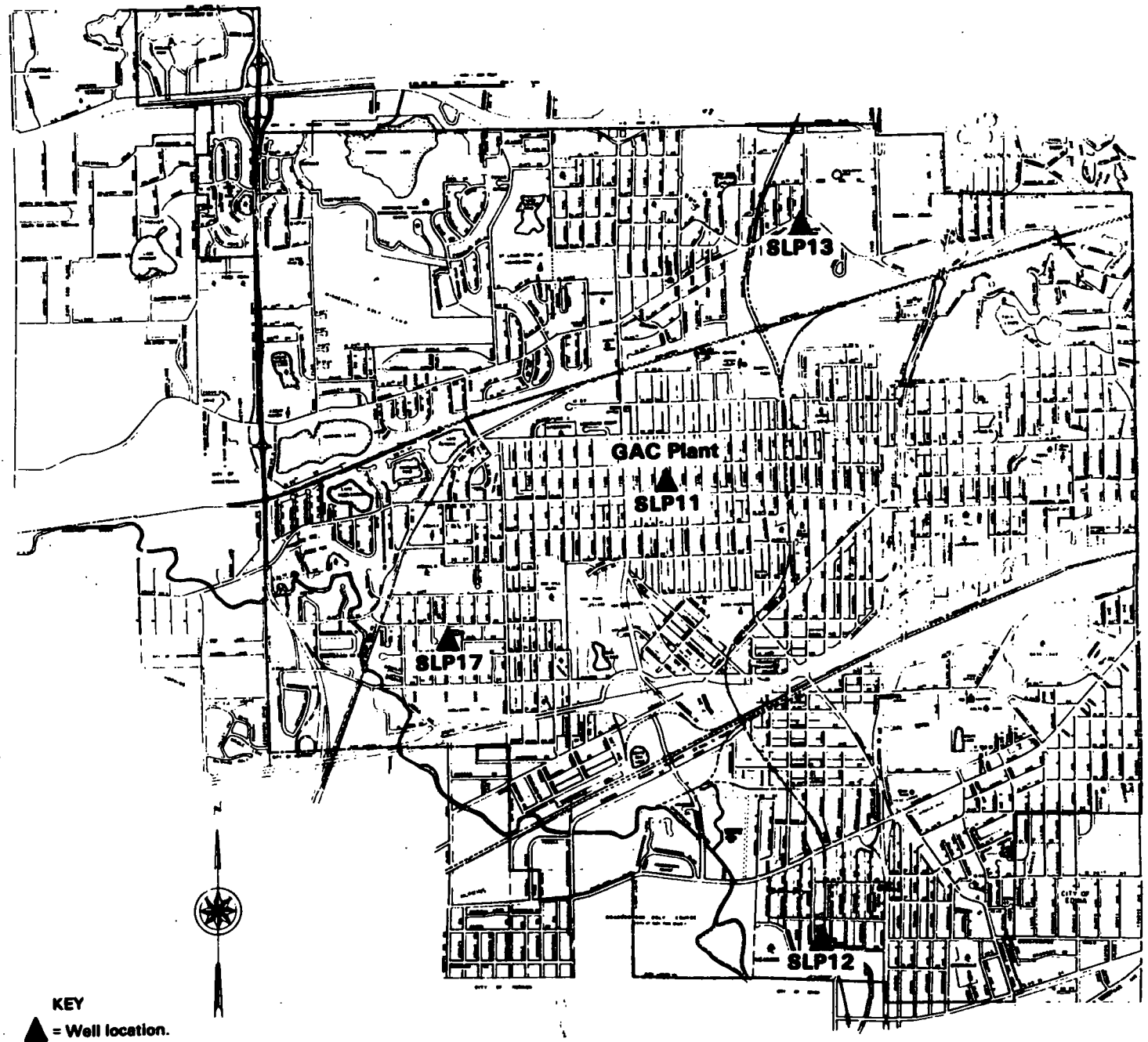


Figure 1 Location of Mt. Simon - Hinkley Monitoring Wells and St. Louis Park GAC Water-treatment Plant



NON-RESPONSIVE

Figure 2 Location of Praire du Chien-Jordan Aquifer Wells

NON-RESPONSIVE

SCALE
0 500 1000

Figure 3 Location of Source and Gradient Control Wells

NON-RESPONSIVE

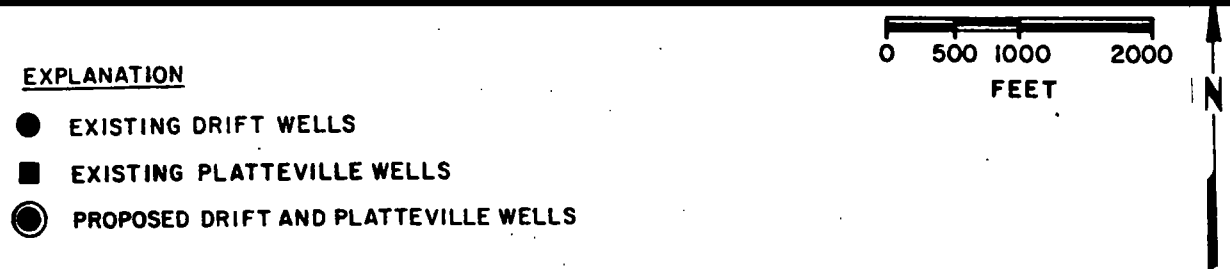


Figure 4 Location of Drift-Platteville Monitoring Wells

NON-RESPONSIVE

EXPLANATION

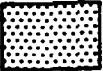
- ▲ W 33 LOCATION AND PROJECT WELL NUMBER
- ▲ OBSERVATION WELL COMPLETED IN ST. PETER AQUIFER
- OBSERVATION WELL COMPLETED IN BASAL ST. PETER CONFINING BED
- NEW ST. PETER MONITORING WELLS
- ◎ WELL IN WHICH WATER LEVELS WERE MONITORED WITH A DIGITAL RECORDER DURING PART OF 1978-81
-  BEDROCK VALLEY/CONTACT WHERE UNCONSOLIDATED DRIFT DEPOSITS OVERLIE ST. PETER SANDSTONE

Figure 5 Proposed and Existing St. Peter Aquifer Well Locations and Bedrock Valley

Identification of Wells to be Monitored

The RAP specifies the majority of wells to be monitored, but leaves the identification of 30 Drift-Platteville Aquifer wells to this Plan. Specifically, Section 9.6 of the RAP requires 30 Drift-Platteville Aquifer monitoring wells to be sampled semi-annually during the first year, annually during the second and third years, thereafter, 20 wells are to be sampled biennially. Thirty candidate Drift-Platteville Aquifer monitoring wells have been chosen including W131, W136, the six new Drift-Platteville Aquifer wells installed for the Northern Area Remedial Investigation, eleven other existing Drift Aquifer monitoring wells, and eleven other Platteville Aquifer monitoring wells (Figure 4). The 22 other existing Drift-Platteville Aquifer monitoring wells selected for monitoring are as follows:

<u>Drift Aquifer Monitoring Wells</u>	<u>Platteville Aquifer Monitoring Wells</u>
W2, W6	W1, W18, W19
W10, W11, W12	W20, W27
W116, W117	W101, W120
W128, W135	W121, W124
P109, P112	W130
	W143

The objectives of monitoring ground-water in the Drift-Platteville Aquifer are to: 1) assess changes in the extent of contamination, and 2) to evaluate the effectiveness of the source and gradient control well systems and any other remedy implemented in the Drift-Platteville Aquifer. In order to address the first objective, Drift-Platteville Aquifer monitoring wells that provide adequate coverage of the area surrounding the contaminant source area were chosen. While only two upgradient or cross-gradient wells were chosen (W1 and W2) most of the selected wells are spread out around the area downgradient from the site and bog contaminant source areas (Figure 4).

The second objective is addressed by selecting wells that will provide both water level and water quality data that will help assess the effectiveness of the source and gradient control well systems. In this regard, the water quality data are judged to be more significant than the water level data, because the purpose of these systems is to control the distribution of contaminants. Therefore, wells outside of the hydraulic influence of a pumping well should reflect the influence of the pumping well on water quality in the aquifer. Also, the pumping wells themselves will be monitored quarterly in accordance with the RAP, and pumping test data will be used to determine the hydraulic influence of the pumping wells. Therefore, the wells shown on Figure 4 are selected primarily for the water quality samples they will provide.

GROUND-WATER SAMPLING PROCEDURES

An important distinction is made between the sampling procedures for active pumping wells (eg. municipal wells) and for non-pumping monitoring wells. Active pumping wells are used on a regular basis, have dedicated pumps and associated plumbing, and have sample taps for collecting samples. Non-pumping monitoring wells may be new, or may have not been pumped for several years, and most require pumping and associated equipment for sampling. Another distinction is that the active pumping monitoring wells are typically located inside buildings whereas monitoring wells are not.

With these considerations in mind, the Initial Sampling Plan has been developed so that the ground-water monitoring program in each aquifer meets the requirements and intent of the RAP. Ground-water monitoring will be conducted in accordance with the procedures given in the Quality Assurance Project Plan (QAPP), and with "Procedures for Ground-Water Monitoring: Minnesota Pollution Control Agency Guidelines", April 1985. Well logs for existing wells that will be monitored, which have not been presented in any other submittal, are given in Appendix B.

Water Level Measurements

Water level measurements will be made using electric tapes or weighted steel tapes. Steel tapes will be used whenever possible because of their generally greater precision compared to electric tapes. Water level measurements using steel tapes will be made by suspending a known length of tape in the well so that the bottom end of the tape is below the water level. The lower portion of tape will be coated with blue chalk that exhibits a noticeable color change when wetted. The water level measurement will be obtained by subtracting the length of wetted tape from the total length of tape suspended below the measuring point of each well.

If reliable water marks on chalked portions of steel tapes can not be discerned because of water on the inside of the well casing or pump discharge pipe, then an electric tape will be used for water level measurements. Using the electric tape, the probe at the end of the tape will be lowered slowly in the well until contact with the water is made. Because of surface tension, readings of the water level made when the probe enters the water will differ from readings made when the probe leaves the water, thus breaking surface tension. To standardize these measurements, the second reading will always be used (i.e., the reading made when the probe leaves the water).

Water level measurement made for the purpose of defining ground-water flow patterns in a particular aquifer will be performed independently from ground-water sampling, as a discrete event

(probably lasting one or two days). The wells will be revisited for sampling, and measurements to determine the volume of water in the well will be made at that time.

Sample Collection at Active Pumping Wells

At active pumping wells the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team.

Water level measurements will then be made, if practical. The normal operation of the well will not be interrupted for the purpose of measuring water levels. An electric tape will be used to measure water levels in pumping wells. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered. The only exception to this is the GAC plant monitoring under RAP Section 4.3 which includes treated-water monitoring.

If it can not be determined that a well has been pumping at some time during the 24 hour period preceding sampling, or if it is known the well was not pumping, then the well shall be purged until field measurements of temperature, pH, and specific conductance have stabilized after at least three well volumes have been removed from the well. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

Sample Collection at Monitoring Wells and Piezometers

Because unanticipated or changed conditions may cause difficulty in the purging and sampling of the monitoring wells and piezometers, flexibility in the approach to sample retrieval is necessary. This Plan proposes that the sampling team be given latitude in the selection of purge/sample equipment and procedures necessary to complete the monitoring task.

Table 2 specifies that Prairie du Chien-Jordan Aquifer monitor well W70, St. Peter Aquifer monitor wells W24 and W33 and Drift-Platteville Aquifer monitor well W117 be monitored. Each well is equipped with a dedicated submersible pump and it will be the responsibility of the sampling team to determine if the pump is operable. In the event the dedicated pump within any individual well is operable, well purging and sample retrieval tasks will be completed with the aid of the pump in conformance with parameter monitoring established herein. In the event the dedicated pump within

any individual well is inoperable, the pump will be removed and purging/sampling procedures will be as established below.

Monitoring wells and piezometers not equipped with dedicated submersible pumps will be purged using a nondedicated submersible pump, suction pump or bailer. During the purging of each well, temperature, pH, and specific conductance of the purge water will be monitored using a Hydrolab water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of these readings will indicate that purging is complete and sampling may commence. Upon completion of well purging, samples will be collected from each well using a stainless steel or teflon bailer and a new length of nylon or polyester rope.

Samples will be collected by filling each of the appropriate sample containers in rapid succession, without prerinsing the containers with sample. The bottle will be held under the sample stream without allowing the mouth of the bottle to come in contact with the bailer and filled completely, and the cap securely tightened. Bottles will be checked for air and if air is visible, the cap removed and more sample added. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

The discharge from purging monitoring wells will be handled in accordance with the Contingency Plan (Appendix C). In general, if a visible sheen can be seen on the water surface, the discharge will be routed to the sanitary sewer. Otherwise, the storm sewer or surface water discharge will be used. Non-dedicated ground-water sampling or monitoring equipment that comes in contact with the ground water will be decontaminated between uses, as described in the QAPP.

ANALYTICAL PROGRAM

Tables 1 and 2 show the ground-water monitoring summary as prescribed in the RAP. Indicated on the tables are the analyses required. Expanded analyses including some priority and conventional pollutants may also be required according to RAP Section 9.3.3. Details of all analytical methodology can be found in the QAPP and its appendices. All analyses will be performed at Rocky Mountain Analytical Laboratory's (RMAL) Arvada, Colorado analytic facility. RMAL has agreed to provide a turnaround time of 30 working days from the receipt of samples to the submittal of analytical reports. The laboratory will notify the City of St. Louis Park if it can not meet this turnaround time.

Ground-water monitoring will include two methods of PAH analyses depending upon the anticipated PAH concentration levels. Low level (nanograms per liter or part per trillion) PAH analyses will be performed utilizing selected ion monitoring gas chromatography mass spectrometry. This method will be used to analyze samples from drinking water wells and from other wells for which the RAP requires drinking water criteria to be enforced (e.g., St. Peter Aquifer monitoring wells). Non-criteria level (micrograms per liter or part per billion) PAH analyses, using EPA Method 625, will be performed on samples from wells that have historically contained elevated PAH concentrations (e.g., part per million levels in wells W23 and W105), and initially on wells that are not subject to the RAP's requirements for meeting drinking water criteria (e.g., Drift-Platteville Aquifer monitoring wells).

Two methods are required for PAH analyses because the low level part per trillion method is not appropriate for samples containing more than approximately 20 micrograms per liter of total PAH. Analysis of samples containing total PAH concentrations over 20 micrograms per liter, if performed with the low level method, requires multiple dilutions and increases the risk of cross-contamination of the samples. This decreases the reliability of the data. Not only will multiple dilutions increase the variability of measurements, but critical quality control information (e.g., surrogate recoveries) is lost. Therefore, for samples containing greater than 20 micrograms per liter of total PAH the analytical method that will be used is EPA Method 625 as described in the Quality Assurance Project Plan (Section 4.6).

The EPA Method 625 analysis will be performed on one-liter samples, and will have detection limits of 10 micrograms per liter. For wells that are tested with this non-criteria method, if the analytical results of the first sampling indicates total PAH concentrations less than 20 micrograms per liter, the low level method will be used to analyze samples from subsequent sampling rounds. This procedure will allow an evaluation of long-term PAH concentrations around the fringe of PAH contamination in the Drift-Platteville Aquifer.

Depending on the circumstances and the actual PAH level, first-round analytical results using the low level method that exceed 20,000 nanograms per liter of total PAH will indicate a switch to EPA Method 625 for subsequent sampling rounds.

REPORTING

The analytical reporting requirements of the Consent Decree and RAP are identified in Part K of the Consent Decree, and Sections 3.4, 4.3.5, 12.1.1, and 12.1.2 of the RAP. Part K requires Reilly to submit annual progress reports on March 15, 1989. This report will contain analytical reports as specified in Section 5.0 of the QAPP for this Initial Sampling Plan, all water level measurements and chemical analyses that have not been presented in previous reports (e.g., the St. Peter Remedial Investigation Report will present all of the data for the St. Peter Aquifer), and interpretive maps and tables, as specified in RAP Section 3.4(B) and (C). Also the effectiveness of the source and gradient control well systems in the Drift-Platteville Aquifer will be discussed in the annual report.

The reporting requirement for each aquifer, and for the GAC treatment plant, are described below.

GAC Treatment Plant

RAP Section 4.3.5 requires the City to submit an annual report that presents the results of all monitoring of the GAC treatment system. Analytical results for wellhead water, feed water, and treated water will be included in this report. The report will also describe briefly the operating performance of the GAC plant during the previous calendar year. The GAC plant annual reports are due each March 15th.

Mt. Simon-Hinckley Aquifer

The monitoring data for the Mt. Simon-Hinckley Aquifer will be included in the annual report. In addition to the results of all water level measurements and chemical analyses, the report will contain a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h)anthracene labelled by the location of each well in accordance with RAP Section 3.4(C). Since the Mt. Simon-Hinckley Aquifer wells are monitored on an annual basis, there will be only one sampling event to report.

Ironton-Galesville Aquifer

The monitoring data for the Ironton-Galesville Aquifer will be included in the annual report. Since well W105 is the only well that will be sampled in this aquifer and only one other well (W38) will be used for water level measurements, the monitoring data will be reported in tabular form as well as in map form as required by RAP Section 3.4.

Prairie du Chien-Jordan Aquifer

The monitoring data for the Prairie du Chien-Jordan Aquifer will be included in the annual report. The results of all water level measurements and chemical analyses will be included. For each of the quarterly measuring periods a water level contour map will be prepared with elevations labelled at each well. For each sampling event, a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h)anthracene labelled by the location of each well will be prepared in accordance with RAP Section 3.4(C).

St. Peter Aquifer

The monitoring data for the St. Peter Aquifer will be reported in the St. Peter Remedial Investigation Report, in accordance with RAP Section 8.1.4. The results of all water level measurements and chemical analyses will be included. For each measuring period in the St. Peter Aquifer, a water level contour map will be prepared with elevations labelled at each well. For each sampling event, a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h)anthracene labelled by the location of each well will be prepared in accordance with RAP Section 3.4(C).

Drift-Platteville Aquifer

The monitoring data for the Drift-Platteville Aquifer will be included in the annual report, and in the Northern Area Remedial Investigation Report. The results of all water level measurements and chemical analyses will be included in both reports. For each measuring period in the Drift-Platteville Aquifer a water level contour map will be prepared with elevations labelled at each well. For each sampling event, a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h)anthracene labelled by the location of each well, and a map with phenolics concentrations labelled by the location of each well will be prepared in accordance with RAP Section 3.4. The Drift-Platteville Aquifer monitoring data will be included in the Northern Area Remedial Investigation Report because of its relevance to providing a further definition of the nature and extent of contamination in the Northern Area. The same data will be included in the annual report to support a discussion of the results with respect to the effectiveness of the source and gradient control well systems.

APPENDIX A
ADDITIONAL MONITORING REQUIREMENTS

Level or Drinking Water Criterion is exceeded during the first year of operation of the system, Reilly shall immediately notify the Regional Administrator, the Director, and the Commissioner, and shall undertake such additional Monitoring as is required by Section 4.3.2.

- (D) Routine Monitoring after two carbon changes shall be quarterly, unless the Regional Administrator, the Director, and the Commissioner determine that the observed service life of the carbon is too short to permit this frequency, in which case the Regional Administrator, the Director and the Commissioner shall notify Reilly of the required Monitoring frequency in accordance with Part G or H of the Consent Decree.

4.3.2. Carbon Replacement Monitoring

- (A) If the analytical results from any treated water sample obtained pursuant to Section 4.3.1. exceed the Drinking Water Criterion for Other PAH or exceed the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then Reilly shall collect two additional treated water samples at least 2 Days apart within one week of receiving the results of the exceedance sample. If the

analytical results from either one or both of the two additional samples also exceed the Drinking Water Criterion for Other PAH or the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, and neither of the conditions specified in (C)(1) and (2) below are met, then the carbon shall be replaced within 21 Days of receiving the additional sample results.

(B) If the analytical results from any treated water sample obtained pursuant to Section 4.3.1. exceed the Advisory Level for Other PAH, then Monitoring of treated water shall be conducted immediately according to Section 12.1. If the results of any two samples required by Section 12.1. exceed the Drinking Water Criterion for Other PAH, and neither of the conditions specified in (C)(1) and (2) below are met, then the carbon shall be replaced within 21 Days of receiving the additional sample results.

(C) If any analytical result from the additional samples taken as required by (A) or (B) above exceeds the Drinking Water Criterion for Other PAH, or the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene during either

- (1) within one year after the carbon treatment system is placed into service or
- (2) within one year after the first carbon change if carbon was changed in the first year of operation of the carbon treatment system,

then Reilly shall conduct the Monitoring program specified in Section 4.6. Reilly shall report the results of the Section 4.6. Monitoring program to the Regional Administrator, the Director and the Commissioner within 7 Days of receiving the analytical data. If the treated water from the carbon treatment system is determined pursuant to Section 4.6. to exceed the Drinking Water Criterion for Other PAH or the Advisory Levels for Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then Reilly shall replace the carbon within 14 Days of making this determination. If the treated water is determined pursuant to Section 4.6. to meet the Drinking Water Criterion for Other PAH and the Advisory Levels for Carcinogenic PAH and the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then normal GAC system operation and Monitoring in accordance with Sections 4.3.1.(B) and

(C) After the first month of operation, Monitoring of feed water shall be performed quarterly until the carbon has been changed twice. If the Regional Administrator, the Director and the Commissioner determine pursuant to Section 4.3.1.(B) that the GAC system is not operating properly, Reilly may, upon receipt of such determination, be required to resume biweekly Monitoring of feed water.

(D) After two carbon changes in the GAC system, feed water shall be Monitored annually.

4.3.4. Extended Monitoring

Treated water from the GAC system shall be sampled and analyzed annually for the extended list of PAH in Part A.2. of Appendix A, using gas chromatography/mass spectroscopy (GC/MS), or other methods approved by the Regional Administrator and the Director. During this extended analysis, any compounds listed in Part A.2. of Appendix A, or any other compounds which are detected with significant peak heights that are not routinely Monitored, shall be identified and, if possible, quantified, using a mass spectral library which contains extensive spectra of PAH compounds, such as the National Bureau of Standards mass spectral library. Reilly shall analyze a sample of treated or feed water once a year for the acid fraction compounds determined by EPA Test Method 625 or by other methods approved by the Regional Administrator and the Director.

approved by the Regional Administrator and Director in accordance with Part G of the Consent Decree.

4.6. Testing of Compliance

During the test period prior to connecting the carbon treatment system to the municipal distribution system, within the first year of operation and within the first year after the first carbon change (if such change occurs during the first year of operation), the determination of whether treated water from the carbon treatment system meets the Drinking Water Criterion for Other PAH and the Advisory Levels for Carcinogenic PAH and the sum of benzo(a)pyrene and dibenz(a,h)anthracene shall be based on the following testing procedures: within ten Days of receiving analytical results of Monitoring pursuant to Section 4.3.2.(C) indicating that special Monitoring pursuant to this Section 4.6 is required, Reilly shall collect at least four and no more than six samples of treated water on at least four and no more than six successive days and shall collect a field blank sample corresponding to each treated water sample. The samples shall be analyzed for Carcinogenic PAH and Other PAH in accordance with procedures developed and approved pursuant to Section 3.2 with analytical results to be provided in 21 Days or less pursuant to Section 2.8. The analytical values so obtained shall be subjected to the following statistical test to determine whether the treated water exceeds the Drinking Water Criterion for Other PAH or the Advisory Levels for Carcinogenic

PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene:

$$\text{if } t_{m,0.95} < \frac{\bar{x}_{s,i} - \bar{x}_{b,i} - C_i}{((S_{s,i}^2 + S_{b,i}^2)/n)^{1/2}}$$

then treated water from the carbon treatment system shall be determined to have exceeded the applicable Drinking Water Criterion or Advisory Levels, where:

n = number of sample events

$t_{m,0.95}$ = Student's t -test statistic for m degrees of freedom and a probability of 0.95

m = the closest integer to the value of:

$$(n-1) \left[1 + \frac{2}{(S_{s,i}/S_{b,i})^2 + (S_{b,i}/S_{s,i})^2} \right]$$

$\bar{x}_{s,i}$ = mean the treated water samples for value i

$\bar{x}_{b,i}$ = mean of the blank samples for value i

$S_{s,i}$ = Standard Deviation of $\bar{x}_{s,i}$

$S_{b,i}$ = Standard Deviation of $\bar{x}_{b,i}$

C_i = Drinking Water Criterion for Other PAH, Advisory Level for Carcinogenic PAH, or Advisory Level for the sum of benzo(a)pyrene and dibenz(a,h)anthracene

i = the sum of Other PAH compounds, the sum of Carcinogenic PAH compounds, or the sum of benzo(a)pyrene and dibenz(a,h)anthracene.

CONTINGENT ACTIONS FOR MUNICIPAL
DRINKING WATER SUPPLY WELLS

12.1. Contingent Monitoring

12.1.1. Exceedance of Advisory Levels

If the analytical result of any sample taken from an active municipal drinking water well under the Monitoring requirements of Sections 3., 4.3., 5.1., 6.2.1., 7.3., or 8.4. above exceeds an Advisory Level, Reilly shall take another sample within seven Days of receiving the analytical results and analyze this sample. If the results of the second sample are below all of the Advisory Levels, a third sample shall be taken by Reilly within seven Days of receiving the results of the second sample. If the third sample is below all of the Advisory Levels, Monitoring of the affected well shall revert to its normal schedule. If the analytical result of the second or third sample exceeds an Advisory Level but is less than all Drinking Water Criteria, the Regional Administrator, the Director, and the Commissioner shall be notified by Reilly immediately and subsequent samples shall be taken by Reilly monthly until such time as either:

- (A) three consecutive samples yield results less than all of the Advisory Levels, in which case the sampling interval shall revert to the level specified for the affected well in Sections 3., 4.3., 5.1., 6.2.1., 7.3., or 8.4. above; or

- (B) a sample yields results greater than a Drinking Water Criterion, in which case the requirements of Section 12.1.2., below, apply.

12.1.2. Exceedance of Drinking Water Criteria

- (A) If the analytical result of any sample taken from an active municipal drinking water well pursuant to Section 12.1.1 exceeds the Drinking Water Criterion for Carcinogenic PAH, the sum of benzo(a)pyrene and dibenz(a,h)anthracene, or Other PAH, the Regional Administrator, the Director and the Commissioner shall be immediately notified by Reilly, and another sample shall be taken by Reilly within three Days of receiving the results of the first sample and analyzed. If the analytical result of the second sample is less than all of the Drinking Water Criteria but greater than any Advisory Level, a third sample shall be taken by Reilly within seven Days of receiving the results of the second sample and analyzed. If the results of this third sample are less than all of the Drinking Water Criteria, but greater than any Advisory Level, Reilly shall comply with the monthly sampling frequency specified in Section 12.1.1. above.

(B) If the analytical result of the second or third sample taken pursuant to Section 12.1.2.(A) above is greater than the Drinking Water Criterion for Carcinogenic PAH, the sum of benzo(a)pyrene and dibenz(a,h)anthracene, or Other PAH, Reilly shall Monitor the well weekly until such time as either: (1) three consecutive samples yield results below all of the Drinking Water Criteria, in which case Monitoring of the well shall revert to the normal schedule (including Advisory Level Monitoring as specified by Section 12.1.1. above if applicable); or, (2) three consecutive samples yield results above any Drinking Water Criterion, in which case Reilly shall immediately notify the Regional Administrator, the Director and the Commissioner. The Commissioner may then require the affected well to be taken out of service, in which case Reilly shall undertake the contingent actions specified in Section 12.2. below.

12.I.3. Analytical Turn-around Time

All Monitoring conducted pursuant to Section 12.1. shall be on a 21-Day turn-around time basis in accordance with Section 2.8.

APPENDIX B

WELL LOGS

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PROVISIONAL RECORDS
Subject to Revision

Table 1.—Data on municipal wells in the St. Louis Park area, Minnesota—Continued

Township and range	Site identification (latitude and longitude)	Minnesota well number	Project well number	Owner name or other identifiers	Driller	Date drilled	Driller's log	Land surface altitude, in feet	Reported depth of well, in feet	Casing diameter and depth, in feet	Aquifer(s) open to well bore	Water level, in feet	Date measured	Field measurement status	Remarks
117N21W1602A1	445615093212301	206443	W 112	Old St. Louis Park well #1	McCarthy	05-32	0-109 Qd 129-274 Qp 274-398 Qp 398-486 Cj	917.5	590	16 in. 0-212 12 in. 194-274	In 1932 Ope-Cd in 1978 Qp	83	2-4-82	Q,M	Also included in table
117N21W1602D1	445701093215801	206441	SLP 1	St. Louis Park municipal well 1	Max Ranner	00-38	0-104 Qd 134-136 Qp 136-236 Qp 236-280 Qp	925	290	16 in. 0-104	Opl-Osp	60	0-0-59	A	
117N21W1602D2	445659093220201	200962	SLP 2	St. Louis Park municipal well 2	do	00-38	0-104 Qd 134-136 Qp 136-236 Qp 236-280 Qp	925	290	16 in. 0-104	Opl-Osp	60	0-0-59	A	
117N21W1602D3	445701093215803	206440	W 113	St. Louis Park municipal well 3	McCarthy	08-39	0-103 Qd 173-118 Qp 118-230 Qp 230-286 Qp	922	286	24 in. 0-103	Opl-Osp	49	2-4-82	P	Latitude is 445702 Also included in table
028N24W078A	445546093202201	200542	SLP 4	St. Louis Park municipal well 4	Layne- Minnesota	09-46	0-89 Qd 89-119 Qp 119-250 Qp 250-290 Qp 290-411 Qp 411-483 Cj 483-503 Cj	900	503	24 in. 0-90 18 in. 0-304	Ope-Cj Cd	85	9-27-46 PROVISIONAL RECORDS Subject to Revision	P	
117N21W1602D1	445611093230301	203106	SLP 5	St. Louis Park municipal well 5	do	10-47	0-109 Qd 129-120 Qp 120-132 Qp 132-230 Qp 230-285 Qp 285-407 Qp 407-465 Cj	930	465	24 in. 0-115 20 in. 0-305	Ope-Cj	109	2-4-82	P	
117N21W1602D1	445520093211801	206457	SLP 6	St. Louis Park municipal well 6	do	03-48	0-90 Qd 90-122 Qp 122-127 Qp 127-190 Qp 190-417 Qp 417-480 Cj	915	480	24 in. 0-107 20 in. 0-303	Ope-Cj	102	2-4-82	P	
117N21W1602D1	445702793221701	206436	SLP 7	St. Louis Park municipal well 7	do	05-52	0-75 Qd 75-97 Qp 97-100 Qp 100-210 Qp 210-260 Qp 260-380 Qp 380-440 Cj 440-446 Cj	905	446	24 in. 0-80 20 in. 0-287	Ope-Cj	87	2-4-82	P	Latitude is 445730
117N22W1601D1	445807093241001	227964	SLP 8	St. Louis Park municipal well 8	Bergeson- Cassell	00-55	0-108 Qd 138-135 Qp 135-132 Qp 132-242 Qp 242-303 Qp 303-413 Qp 413-495 Cj 495-507 Cj	940	507	24 in. 0-253 16 in. 0-343	Cj-Cd	130	2-4-82	P	Longitude is 0932008
117N21W1602D2	445730093222201	206437	SLP 9	St. Louis Park municipal well 9	Layne- Minnesota	06-56	0-69 Qd 69-120 Qp 120-220 Qp 220-275 Qp 275-380 Qp 380-473 Cj	905	473	24 in. 0-81 16 in. 0-289	Ope-Cj	70	6-6-56	P	
117N21W1602D4	445700093221304	206442	SLP 10	St. Louis Park municipal well 10	Kays	09-55	0-105 Qd 105-125 Qp 125-290 Qp 290-409 Qp 409-500 Cj	926	500	24 in. 0-106 16 in. 0-315	Ope-Cj	111	2-4-82	P	Latitude is 445702 Longitude is 0932204

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Table 1.—Data on municipal wells in the St. Louis Park area, Minnesota—Continued

Township and range	Site identification (latitude and longitude)	Minnesota unique well number	ISDS project well number	Owner name or other identifiers	Driller	Date drilled	Driller's log	Land surface altitude, in feet	Reported depth of well, in feet	Casing diameter and depth	Aquifer(s) open to well bore	Water level, in feet	Date measured	Field measurement status	Remarks
117N21W08R05	445703093220201	206439	SLP 11	St. Louis Park municipal well 11	Bergeson-Caswell	11-60	0-101 101-120 120-288 288-408 408-505 505-530 530-683 683-745 745-853 853-955 955-1093	920	1093	24 in. 16 in. 10-880	Qm-Ch	258	2-4-82	P	Latitude is 445704
117N21W08R02	445526093211402	206456	SLP 12	St. Louis Park municipal well 12	Keys	10-65	0-96 96-127 127-132 132-202 202-427 427-505 505-550 550-695 695-725 725-745 745-812 812-883 883-1095	915	1095	30 in. 0-99 24 in. 0-270 16 in. 0-900	Qm-Ch	245	10-65	P	Latitude is 445523 Longitude is 0932112
117N21W08R01	445754093212201	206424	SLP 13	St. Louis Park municipal well 13	Layne-Minnesota	07-64	0-94 94-101 101-212 212-270 270-386 386-460 460-490 490-655 655-714 714-770 770-917 917-1040 1040-1045	900	1040	30 in. 0-95 24 in. 0-212 16 in. 0-891	Qm-Psh	255	07-64	P	Longitude is 0932123

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117N21W08R02	445513093213602	227965	SLP 14	St. Louis Park municipal well 14	Bergeson-Caswell	02-65	0-94 94-98 98-365 365-375 375-475 475-485	900	485	30 in. 0-94 24 in. 0-253 16 in. 0-389	CJ	89	2-4-82	P	Latitude is 445754 Longitude is 0932123
117N21W08R06	445703093220202	215447	SLP 15	St. Louis Park municipal well 15	Bergeson-Caswell	00-69	0-102 102-124 124-288 288-402 402-482 482-503	925	503	30 in. 0-102 24 in. 0-402	CJ	110	2-4-82	P	Latitude is 445704
117N21W07E8A	445750093234801	203187	SLP 16	St. Louis Park municipal well 16	Tri-State	07-73	0-105 105-128 128-258 258-294 294-310 310-495 495-500	920	500	50 in. 0-310 24 in. 0-425	CJ	120	2-4-82	P	
117N21W18R0B3	445631093230303	—	SLP 17	St. Louis Park municipal well 17	Layne-Minnesota		0-95 95-120 120-210 210-270 270-390 390-470 470-760	925	760	16 in. 0-95 12 in. 0-286	Qm-CJ Cg	—	—	P	Presently under construction
117N22W02E8A	445514093240301	206573	HOP 1	Hopkins municipal well 1	McCarthy	00-20	0-95 95-120 120-210 210-270 270-390 390-470 470-760	925	760	16 in. 0-95 12 in. 0-286	Qm-CJ Cg	—	—	P	

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Table — Data on municipal wells in the St. Louis Park area, Minnesota—Continued

Township and range	Site identification (latitude and longitude)	Unique well number	Project number	Owner name or other identifiers	Driller	Date drilled	Driller's log	Land surface altitude, in feet	Reported depth of well, in feet	Casing diameter and depth, in feet	Aquifer(s) open to well bore	Water level, in feet	Date measured	Field measurement status	Remarks
117N22W24R2E	445509524001	207995	HDP 2	Hopkins municipal well 2	McCarthy	00-34	0-87 (M) 37-117 (M) 117-220 (M) 230-299 (M) 299-405 (M) 405-475 (M) 475-680 (M) 680-720 (M) 720-728 (M)	920	728	16 in. 0-89 12 in. 0-202	Op-Clg	72	1948	AR3	
117N22W19R4E	44554708230001	208108	HDP 3	Hopkins municipal well 3	do	00-48	0-80 (M) 30-110 (M) 110-116 (M) 116-225 (M) 225-280 (M) 280-460 (M) 460-475 (M)	920	475	24 in. 0-86 20 in. 0-292 16 in. 0-132	Op-CJ	43	3-3-82	P	
117N22W13R3E	44550709320501	204068	HDP 4	Hopkins municipal well 4	Bergerson-Caswell	05-54	0-134 (M) 134-171 (M) 171-175 (M) 175-339 (M) 339-450 (M) 450-530 (M) 530-548 (M)	980	548	—	—	164	3-3-82	P	
117N22W20R2E	44550209220501	204570	HDP 5	Hopkins municipal well 5	do	05-67	0-179 (M) 179-260 (M) 260-317 (M) 317-427 (M) 427-489 (M) 489-500 (M)	967	500	30 in. 0-240 24 in. 217-3204 16 in. 0-3824	Op-CJ	141	3-3-82	P	
117N22W24R2E	44550709324001	112228	HDP 6	Hopkins municipal well 6	do	09-77	0-134 (M) 134-165 (M) 165-281 (M) 281-333 (M) 333-356 (M) 356-545 (M)	945	545	30 in. 0-132 24 in. 0-354	Op-CJ	150	3-3-82	P	Longitude is 0932438
117N22W22R3E	44551609220502	208011	MUK 1	Munnetonka municipal well 1	do	08-55	0-229 (M) 229-269 (M) 269-407 (M) 407-513 (M)	1045	513	—	—	—	—	AR	
117N22W24R2E	44551109220401	208110	MUK 2	Munnetonka municipal well 2	Tri-State	03-62	0-251 (M) 251-270 (M) 270-384 (M) 384-427 (M) 427-530 (M) 530-619 (M)	—	619	16 in. 0-265 10 in. 0-545	CJ	220	3-62	P	
117N22W20R2E	44554909323701	204470	MUK 3	Munnetonka municipal well 3	Tri-State	10-63	0-118 (M) 118-247 (M) 247-378 (M) 378-461 (M) 461-465 (M)	921	465	24 in. 0-123 20 in. 0-196 18 in. 0-393	CJ	39	10-63	P	
117N22W22E		171021	MUK 3A	Munnetonka municipal well 3A	E. H. Renner	03-81	0-105 (M) 105-120 (M) 120-195 (M) 195-250 (M) 250-382 (M) 382-465 (M) 465-468 (M)	—	468	30 in. 0-129 24 in. 0-1864 16 in. 0-254	Op-CJ	53	01-81	P	
117N22W20R2E	44542408230001	205082	MUK 4	Munnetonka municipal well 4	Bergerson-Caswell	09-57	0-172 (M) 172-315 (M) 315-438 (M) 438-530 (M)	960	530	—	—	100	9-16-57	—	
117N22W20R2E	44541009320101	205133	MUK 5	Munnetonka municipal well 5	do	12-56	0-263 (M) 263-279 (M) 279-338 (M) 338-460 (M) 460-545 (M) 545-581 (M)	1000	581	—	—	131	12-12-56	—	

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Table —Data on municipal wells in the St. Louis Park area, Minnesota—Continued

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Township and range	Site identification (latitude and longitude)	Minnesota unique well number	USGS project well number	Owner name or other identifiers	Driller	Date drilled	Driller's log	Land surface altitude, in feet	Reported depth of well in feet	Casing diameter and depth	Aquifer(s) open to well bore	Water level, in feet	Date measured	Field measurement status	Remarks
117N23W12E1C1	445703093245401	204054	MIK 6	Minnnetonka municipal well 6	Keys	06-67	0-101 : d 101-117 : pl 117-133 : gl 133-248 : ap 248-310 : apl 310-392 : pe 392-486 : CJ 486-488 : d1	—	488	24 in. 0-103 20 in. 0-320 16 in. 312-394	CJ	—	—	—	
117N23W12E1C2	445703093245402	208012	MIK 7	Minnnetonka municipal well 7	do	07-67	0-104 : d 104-112 : d 112-121 : pl 121-136 : gl 136-249 : ap 249-304 : apl 304-392 : pe 392-485 : CJ 485-486 : d1	—	486	24 in. 0-114 20 in. 0-315 16 in. 299-341 12 in. 284-397	CJ				
117N23W12E1CA	445538093260101	208013	MIK 8	Minnnetonka municipal well 8	—	00-58	0-165 : d 165-188 : pl 188-339 : ap 339-471 : pe 471-559 : CJ 559-564 : d1	—	564	8 in. 0-170 6 in. 160-378	Ope-CJ	116	00-00	AR	
117N23W12E1CB	445747093272701	204008	MIK 9	Minnnetonka municipal well 9	E. H. Renner	08-68	0-124 : d 124-140 : pl 140-150 : gl 150-250 : ap 250-305 : apl 305-305 : pe	960	395	10 in. 0-312	Ope	100	8-68	AR	
<div> <div>PROVISIONAL RECORDS</div> <div>Subject to Revision</div> <div>U.S. GEOLOGICAL SURVEY WATER RESOURCES DIVISION 702 POST OFFICE BUILDING ST. PAUL, MINNESOTA 55101</div> </div>															
117N23W16E1D1	445634093274501	204140	MIK 10	Minnnetonka municipal well 10	Layne-Minnesota	10-69	0-91 : d 91-126 : pl 126-128 : gl 128-230 : ap 230-292 : apl 292-420 : pe 420-5-5 : CJ	940	505	24 in. 0-112 20 in. 0-235 16 in. 0-305	Ope-CJ	85	10-8-69	P	
<div> <div>PROVISIONAL RECORDS</div> <div>Subject to Revision</div> <div>U.S. GEOLOGICAL SURVEY WATER RESOURCES DIVISION 702 POST OFFICE BUILDING ST. PAUL, MINNESOTA 55101</div> </div>															
117N23W16E1D2	445635093274501	150356	MIK 10A	Minnnetonka municipal well 10A	do	03-81	0-90 : d 90-100 : pl 100-124 : gl 124-132 : ap 132-223 : apl 223-287 : pe 287-405 : CJ 405-481 : d1 481-486 : d1	925	486	30 in. 0-106 24 in. 0-254 16 in. 0-302	Ope-CJ	85	2-25-81	—	
117N23W12E1COC	445445093274201	208014	MIK 11	Minnnetonka municipal well 11	Bergerson-Caswell	00-70	0-181 : d 181-210 : ap 210-272 : apl 272-401 : pe 401-495 : CJ 495-498 : d1	—	498	24 in. 0-184 16 in. 0-282	Ope-CJ	83	00-707		
117N23W10E1D	445808093265001	203717	MIK 12	Minnnetonka municipal well 12	E. H. Renner	06-71	0-130 : d 130-163 : pl 163-168 : gl 158-326 : ap 326-465 : pe 455-532 : CJ 532-535 : d1	—	535	24 in. 0- 16 in. 0-332	Ope-CJ	115	6-71	P	
<div> <div>PROVISIONAL RECORDS</div> <div>Subject to Revision</div> <div>U.S. GEOLOGICAL SURVEY WATER RESOURCES DIVISION 702 POST OFFICE BUILDING ST. PAUL, MINNESOTA 55101</div> </div>															
117N23W13E1A1	445353093260501	205165	MIK 13	Minnnetonka municipal well 13	Layne-Minnesota	04-72	0-234 : d 234-273 : apl 273-398 : pe 398-475 : CJ	—	475	24 in. 0-239 16 in. 0-292	Ope-CJ	—	—	P	
<div> <div>PROVISIONAL RECORDS</div> <div>Subject to Revision</div> <div>U.S. GEOLOGICAL SURVEY WATER RESOURCES DIVISION 702 POST OFFICE BUILDING ST. PAUL, MINNESOTA 55101</div> </div>															
117N23W13E1A2	445353093260502	132263	MIK 13A	Minnnetonka municipal well 13A	Hydro Engineering	06-78	0-193 : d 133-229 : ap 229-258 : apl 258-382 : pe 312-464 : CJ	—	464	24 in. 0-230 16 in. 0-274	Ope-CJ	98	00-00	P	

Table 1—Data on municipal wells in the St. Louis Park area, Minnesota—Continued

Well number	Well name	Owner name or other identifier	Driller	Date drilled	Driller's log	Land surface altitude, in feet	Reported depth of well, in feet	Casing diameter, in feet and depth	Aquifer(s) open to well bore	Water level, in feet	Date measured	Field measurement status	Remarks
117W22422B01	MNK 14	Minnetonka municipal well 14	E. H. Renner	05-72	0-172 172-195 195-204 204-305 305-360 360-490 490-555 CJ	84	555	24 in. 0-177 16 in. 0-367	Op-CJ	175	00-00	P	
PROVISIONAL RECORDS Subject to Revision													
117W22422B02	MNK 14A	Minnetonka municipal well 14A	do	05-78	0-194 195-212 212-219 219-360 360-500 500-570 570-575 CJ	84	575	24 in. 0-196 20 in. 0-343 16 in. 0-395	Op-CJ	195	11-22-78	P	
117W22422B03	MNK 15	Minnetonka municipal well 15	do	02-74	0-183 183-225 225-365 365-490 CJ	85	490	24 in. 0-192 16 in. 0-295	Op-CJ	35	00-00	P	
117W22422B04	MNK 15A	Minnetonka municipal well 15A	Layne-Minnetonka	04-78	0-185 185-227 227-350 350-444 CJ	—	444	24 in. 0-188 16 in. 0-298	Op-CJ	27	4-14-78	P	
028W23018A12	EN 1	Edina municipal well 1	—	1934	0-61 61-90 90-95 95-258 258-385 385-490 490-460 CJ	—	460	16 in. 0-61 12 in. 61-298	Op-CJ	—	—	AR	
028W23018A11	EN 2	Edina municipal well 2	—	1938	0-62 62-97 97-262 262-385 385-391 CJ	—	391	24 in. 0-62 16 in. 0-180	Op-Op	—	—	P	
028W23018B08	EN 3	Edina municipal well 3	—	1949	—	—	422	—	—	76	2-2-82	P	
028W23018B03	EN 4	Edina municipal well 4	Bergeson-Caswell	1950	0-80 80-110 110-270 270-410 410-500 CJ	90	500	24 in. 0-80 16 in. 0-270	Op-CJ	80	—	P	
028W23018B05	EN 5	Edina municipal well 5	Keys	1954	0-200 200-237 237-365 365-443 CJ	873	443	24 in. 0-136 20 in. 0-200 16 in. 0-257	Op-CJ	75	—	P	
028W23018B06	EN 6	Edina municipal well 6	do	06-54	0-24 24-110 110-118 118-278 278-408 408-493 493-503 CJ	90	503	24 in. 0-24 16 in. 0-964 16 in. 0-316	Op-CJ	90	—	P	
117W21262B03	EN 7	Edina municipal well 7	do	03-55	0-132 132-159 159-162 162-230 230-324 324-453 453-545 545-547 CJ	950	547	24 in. 0-132 16 in. 0-350	Op-CJ	112	—	P	
116W21402B08	EN 8	Edina municipal well 8	Bergeson-Caswell	1953	0-220 220-364 364-465 465-472 CJ	870	472	24 in. 0-219 16 in. 0-232	Op-CJ	89	—	P	
PROVISIONAL RECORDS Subject to Revision													

U.S. GEOLOGICAL SURVEY
WATER RESOURCES DIVISION
770 POST OFFICE BUILDING
ST. PAUL, MINNESOTA 55101

Table 1. Data on municipal wells in the St. Louis Park area, Minnesota—Continued

U.S. GEOLOGICAL SURVEY
WATER RESOURCES DIVISION
770 POST OFFICE BUILDING
ST. PAUL, MINNESOTA 55101

413863

Township and range	Site identification (latitude and longitude)	Minnesota unique well number	UNCS project well number	Owner name or other identifier	Driller	Date drilled	Driller's log	Land surface altitude, in feet	Reported depth of well, in feet	Casing diameter and depth, in feet	Aquifer(s) open to well bore	Water level, in feet	Measurement status	Remarks
117N24W32AD	445347003213901	206588	EN 9	Edina municipal well 9	Keys	10-57	0-121 0d 121-131 0d 131-135 0d 135-203 0d 203-213 0d 213-215 0d 215-225 0d 225-270 0d 270-280 0d 280-285 0d 285-289 0d 289-292 0d 292-293 0d 293-294 0d 294-295 0d 295-296 0d 296-297 0d 297-298 0d 298-299 0d 299-300 0d 300-301 0d 301-302 0d 302-303 0d 303-304 0d 304-305 0d 305-306 0d 306-307 0d 307-308 0d 308-309 0d 309-310 0d 310-311 0d 311-312 0d 312-313 0d 313-314 0d 314-315 0d 315-316 0d 316-317 0d 317-318 0d 318-319 0d 319-320 0d 320-321 0d 321-322 0d 322-323 0d 323-324 0d 324-325 0d 325-326 0d 326-327 0d 327-328 0d 328-329 0d 329-330 0d 330-331 0d 331-332 0d 332-333 0d 333-334 0d 334-335 0d 335-336 0d 336-337 0d 337-338 0d 338-339 0d 339-340 0d 340-341 0d 341-342 0d 342-343 0d 343-344 0d 344-345 0d 345-346 0d 346-347 0d 347-348 0d 348-349 0d 349-350 0d 350-351 0d 351-352 0d 352-353 0d 353-354 0d 354-355 0d 355-356 0d 356-357 0d 357-358 0d 358-359 0d 359-360 0d 360-361 0d 361-362 0d 362-363 0d 363-364 0d 364-365 0d 365-366 0d 366-367 0d 367-368 0d 368-369 0d 369-370 0d 370-371 0d 371-372 0d 372-373 0d 373-374 0d 374-375 0d 375-376 0d 376-377 0d 377-378 0d 378-379 0d 379-380 0d 380-381 0d 381-382 0d 382-383 0d 383-384 0d 384-385 0d 385-386 0d 386-387 0d 387-388 0d 388-389 0d 389-390 0d 390-391 0d 391-392 0d 392-393 0d 393-394 0d 394-395 0d 395-396 0d 396-397 0d 397-398 0d 398-399 0d 399-400 0d 400-401 0d 401-402 0d 402-403 0d 403-404 0d 404-405 0d 405-406 0d 406-407 0d 407-408 0d 408-409 0d 409-410 0d 410-411 0d 411-412 0d 412-413 0d 413-414 0d 414-415 0d 415-416 0d 416-417 0d 417-418 0d 418-419 0d 419-420 0d 420-421 0d 421-422 0d 422-423 0d 423-424 0d 424-425 0d 425-426 0d 426-427 0d 427-428 0d 428-429 0d 429-430 0d 430-431 0d 431-432 0d 432-433 0d 433-434 0d 434-435 0d 435-436 0d 436-437 0d 437-438 0d 438-439 0d 439-440 0d 440-441 0d 441-442 0d 442-443 0d 443-444 0d 444-445 0d 445-446 0d 446-447 0d 447-448 0d 448-449 0d 449-450 0d 450-451 0d 451-452 0d 452-453 0d 453-454 0d 454-455 0d 455-456 0d 456-457 0d 457-458 0d 458-459 0d 459-460 0d 460-461 0d 461-462 0d 462-463 0d 463-464 0d 464-465 0d 465-466 0d 466-467 0d 467-468 0d 468-469 0d 469-470 0d 470-471 0d 471-472 0d 472-473 0d 473-474 0d 474-475 0d 475-476 0d 476-477 0d 477-478 0d 478-479 0d 479-480 0d 480-481 0d 481-482 0d 482-483 0d 483-484 0d 484-485 0d 485-486 0d 486-487 0d 487-488 0d 488-489 0d 489-490 0d 490-491 0d 491-492 0d 492-493 0d 493-494 0d 494-495 0d 495-496 0d 496-497 0d 497-498 0d 498-499 0d 499-500 0d 500-501 0d 501-502 0d 502-503 0d 503-504 0d 504-505 0d 505-506 0d 506-507 0d 507-508 0d 508-509 0d 509-510 0d 510-511 0d 511-512 0d 512-513 0d 513-514 0d 514-515 0d 515-516 0d 516-517 0d 517-518 0d 518-519 0d 519-520 0d 520-521 0d 521-522 0d 522-523 0d 523-524 0d 524-525 0d 525-526 0d 526-527 0d 527-528 0d 528-529 0d 529-530 0d 530-531 0d 531-532 0d 532-533 0d 533-534 0d 534-535 0d 535-536 0d 536-537 0d 537-538 0d 538-539 0d 539-540 0d 540-541 0d 541-542 0d 542-543 0d 543-544 0d 544-545 0d 545-546 0d 546-547 0d 547-548 0d 548-549 0d 549-550 0d 550-551 0d 551-552 0d 552-553 0d 553-554 0d 554-555 0d 555-556 0d 556-557 0d 557-558 0d 558-559 0d 559-560 0d 560-561 0d 561-562 0d 562-563 0d 563-564 0d 564-565 0d 565-566 0d 566-567 0d 567-568 0d 568-569 0d 569-570 0d 570-571 0d 571-572 0d 572-573 0d 573-574 0d 574-575 0d 575-576 0d 576-577 0d 577-578 0d 578-579 0d 579-580 0d 580-581 0d 581-582 0d 582-583 0d 583-584 0d 584-585 0d 585-586 0d 586-587 0d 587-588 0d 588-589 0d 589-590 0d 590-591 0d 591-592 0d 592-593 0d 593-594 0d 594-595 0d 595-596 0d 596-597 0d 597-598 0d 598-599 0d 599-600 0d 600-601 0d 601-602 0d 602-603 0d 603-604 0d 604-605 0d 605-606 0d 606-607 0d 607-608 0d 608-609 0d 609-610 0d 610-611 0d 611-612 0d 612-613 0d 613-614 0d 614-615 0d 615-616 0d 616-617 0d 617-618 0d 618-619 0d 619-620 0d 620-621 0d 621-622 0d 622-623 0d 623-624 0d 624-625 0d 625-626 0d 626-627 0d 627-628 0d 628-629 0d 629-630 0d 630-631 0d 631-632 0d 632-633 0d 633-634 0d 634-635 0d 635-636 0d 636-637 0d 637-638 0d 638-639 0d 639-640 0d 640-641 0d 641-642 0d 642-643 0d 643-644 0d 644-645 0d 645-646 0d 646-647 0d 647-648 0d 648-649 0d 649-650 0d 650-651 0d 651-652 0d 652-653 0d 653-654 0d 654-655 0d 655-656 0d 656-657 0d 657-658 0d 658-659 0d 659-660 0d 660-661 0d 661-662 0d 662-663 0d 663-664 0d 664-665 0d 665-666 0d 666-667 0d 667-668 0d 668-669 0d 669-670 0d 670-671 0d 671-672 0d 672-673 0d 673-674 0d 674-675 0d 675-676 0d 676-677 0d 677-678 0d 678-679 0d 679-680 0d 680-681 0d 681-682 0d 682-683 0d 683-684 0d 684-685 0d 685-686 0d 686-687 0d 687-688 0d 688-689 0d 689-690 0d 690-691 0d 691-692 0d 692-693 0d 693-694 0d 694-695 0d 695-696 0d 696-697 0d 697-698 0d 698-699 0d 699-700 0d 700-701 0d 701-702 0d 702-703 0d 703-704 0d 704-705 0d 705-706 0d 706-707 0d 707-708 0d 708-709 0d 709-710 0d 710-711 0d 711-712 0d 712-713 0d 713-714 0d 714-715 0d 715-716 0d 716-717 0d 717-718 0d 718-719 0d 719-720 0d 720-721 0d 721-722 0d 722-723 0d 723-724 0d 724-725 0d 725-726 0d 726-727 0d 727-728 0d 728-729 0d 729-730 0d 730-731 0d 731-732 0d 732-733 0d 733-734 0d 734-735 0d 735-736 0d 736-737 0d 737-738 0d 738-739 0d 739-740 0d 740-741 0d 741-742 0d 742-743 0d 743-744 0d 744-745 0d 745-746 0d 746-747 0d 747-748 0d 748-749 0d 749-750 0d 750-751 0d 751-752 0d 752-753 0d 753-754 0d 754-755 0d 755-756 0d 756-757 0d 757-758 0d 758-759 0d 759-760 0d 760-761 0d 761-762 0d 762-763 0d 763-764 0d 764-765 0d 765-766 0d 766-767 0d 767-768 0d 768-769 0d 769-770 0d 770-771 0d 771-772 0d 772-773 0d 773-774 0d 774-775 0d 775-776 0d 776-777 0d 777-778 0d 778-779 0d 779-780 0d 780-781 0d 781-782 0d 782-783 0d 783-784 0d 784-785 0d 785-786 0d 786-787 0d 787-788 0d 788-789 0d 789-790 0d 790-791 0d 791-792 0d 792-793 0d 793-794 0d 794-795 0d 795-796 0d 796-797 0d 797-798 0d 798-799 0d 799-800 0d 800-801 0d 801-802 0d 802-803 0d 803-804 0d 804-805 0d 805-806 0d 806-807 0d 807-808 0d 808-809 0d 809-810 0d 810-811 0d 811-812 0d 812-813 0d 813-814 0d 814-815 0d 815-816 0d 816-817 0d 817-818 0d 818-819 0d 819-820 0d 820-821 0d 821-822 0d 822-823 0d 823-824 0d 824-825 0d 825-826 0d 826-827 0d 827-828 0d 828-829 0d 829-830 0d 830-831 0d 831-832 0d 832-833 0d 833-834 0d 834-835 0d 835-836 0d 836-837 0d 837-838 0d 838-839 0d 839-840 0d 840-841 0d 841-842 0d 842-843 0d 843-844 0d 844-845 0d 845-846 0d 846-847 0d 847-848 0d 848-849 0d 849-850 0d 850-851 0d 851-852 0d 852-853 0d 853-854 0d 854-855 0d 855-856 0d 856-857 0d 857-858 0d 858-859 0d 859-860 0d 860-861 0d 861-862 0d 862-863 0d 863-864 0d 864-865 0d 865-866 0d 866-867 0d 867-868 0d 868-869 0d 869-870 0d 870-871 0d 871-872 0d 872-873 0d 873-874 0d 874-875 0d 875-876 0d 876-877 0d 877-878 0d 878-879 0d 879-880 0d 880-881 0d 881-882 0d 882-883 0d 883-884 0d 884-885 0d 885-886 0d 886-887 0d 887-888 0d 888-889 0d 889-890 0d 890-891 0d 891-892 0d 892-893 0d 893-894 0d 894-895 0d 895-896 0d 896-897 0d 897-898 0d 898-899 0d 899-900 0d 900-901 0d 901-902 0d 902-903 0d 903-904 0d 904-905 0d 905-906 0d 906-907 0d 907-908 0d 908-909 0d 909-910 0d 910-911 0d 911-912 0d 912-913 0d 913-914 0d 914-915 0d 915-916 0d 916-917 0d 917-918 0d 918-919 0d 919-920 0d 920-921 0d 921-922 0d 922-923 0d 923-924 0d 924-925 0d 925-926 0d 926-927 0d 927-928 0d 928-929 0d 929-930 0d 930-931 0d 931-932 0d 932-933 0d 933-934 0d 934-935 0d 935-936 0d 936-937 0d 937-938 0d 938-939 0d 939-940 0d 940-941 0d 941-942 0d 942-943 0d 943-944 0d 944-945 0d 945-946 0d 946-947 0d 947-948 0d 948-949 0d 949-950 0d 950-951 0d 951-952 0d 952-953 0d 953-954 0d 954-955 0d 955-956 0d 956-957 0d 957-958 0d 958-959 0d 959-960 0d 960-961 0d 961-962 0d 962-963 0d 963-964 0d 964-965 0d 965-966 0d 966-967 0d 967-968 0d 968-969 0d 969-970 0d 970-971 0d 971-972 0d 972-973 0d 973-974 0d 974-975 0d 975-976 0d 976-977 0d 977-978 0d 978-979 0d 979-980 0d 980-981 0d 981-982 0d 982-983 0d 983-984 0d 984-985 0d 985-986 0d 986-987 0d 987-988 0d 988-989 0d 989-990 0d 990-991 0d 991-992 0d 992-993 0d 993-994 0d 994-995 0d 995-996 0d 996-997 0d 997-998 0d 998-999 0d 999-1000 0d	880	1130	24 in. 16 in. 10 in. 533-1010	pch	215	10-57	
028N24W32AD	445153093202101	206184	EN 10	Edina municipal well 10	Bergeson-Cannell	10-63	0-183 0d 183-203 0d 203-209 0d 209-216 0d 216-222 0d 222-226 0d 226-228 0d 228-230 0d 230-232 0d 232-234 0d 234-236 0d 236-238 0d 238-240 0d 240-242 0d 242-244 0d 244-246 0d 246-248 0d 248-250 0d 250-252 0d 252-254 0d 254-256 0d 256-258 0d 258-260 0d 260-262 0d 262-264 0d 264-266 0d 266-268 0d 268-270 0d 270-272 0d 272-274 0d 274-276 0d 276-278 0d 278-280 0d 280-282 0d 282-284 0d 284-286 0d 286-288 0d 288-290 0d 290-292 0d 292-294 0d 294-296 0d 296-298 0d 298-300 0d 300-302 0d 302-304 0d 304-306 0d 306-308 0d 308-310 0d 310-312 0d 312-314 0d 314-316 0d 316-318 0d 318-320 0d 320-322 0d 322-324 0d 324-326 0d 326-328 0d 328-330 0d 330-332 0d 332-334 0d 334-336 0d 336-338 0d 338-340 0d 340-342 0d 342-344 0d 344-346 0d 346-348 0d 348-350 0d 350-352 0d 352-354 0d 354-356 0d 356-358 0d 358-360 0d 360-362 0d 362-364 0d 364-366 0d 366-368 0d 368-370 0d 370-372 0d 372-374 0d 374-376 0d 376-378 0d 378-380 0d 380-382 0d 382-384 0d 384-386 0d 386-388 0d 388-390 0d 390-392 0d 392-394 0d 394-396 0d 396-398 0d 398-400 0d 400-402 0d 402-404 0d 404-406 0d 406-408 0d 408-410 0d 410-412 0d 412-414 0d 414-416 0d 416-418 0d 418-420 0d 420-422 0d 422-424 0d 424-426 0d 426-428 0d 428-430 0d 430-432 0d 432-434 0d 434-436 0d 436-438 0d 438-440 0d 440-442 0d 442-444 0d 444-446 0d 446-448 0d 448-450 0d 450-452 0d 452-454 0d 454-456 0d 456-458 0d 458-460 0d 460-462 0d 462-464 0d 464-466 0d 466-468 0d 468-470 0d 470-472 0d 472-474 0d 474-476 0d 476-478 0d 478-480 0d 480-482 0d 482-484 0d 484-486 0d 486-488 0d 488-490 0d 490-492 0d 492-494 0d 494-496 0d 496-498 0d 498-500 0d 500-502 0d 502-504 0d 504-506 0d 506-508 0d 508-510 0d 510-512 0d 512-514 0d 514-516 0d 516-518 0d 518-520 0d 520-522 0d 522-524 0d 524-526 0d 526-528 0d 528-530 0d 530-532 0d 532-534 0d 534-536 0d 536-538 0d 538-540 0d 540-542 0d 542-544 0d 544-546 0d 546-548 0d 548-550 0d 550-552 0d 552-554 0d 554-556 0d 556-558 0d 558-560 0d 560-562 0d 562-564 0d 564-566 0d 566-568 0d 568-570 0d 570-572 0d 572-574 0d 574-576 0d 576-578 0d 578-580 0d 580-582 0d 582-584 0d 584-586 0d 586-588 0d 588-590 0d 590-592 0d 592-594 0d 594-596 0d 596-598 0d 598-600 0d 600-602 0d 602-604 0d 604-606 0d 606-608 0d 608-610 0d 610-612 0d 612-614 0d 614-616 0d 616-618 0d 618-620 0d 620-622 0d 622-624 0d 624-626 0d 626-628 0d 628-630 0d 630-632 0d 632-634 0d 634-636 0d 636-638 0d 638-640 0d 640-642 0d 642-644 0d 644-646 0d 646-648 0d 648-650 0d 650-652 0d 652-654 0d 654-656 0d 656-658 0d 658-660 0d 660-662 0d 662-664 0d 664-666 0d 666-668 0d 668-670 0d 670-672 0d 672-674 0d 674-676 0d 676-678 0d 678-680 0d 680-682 0d 682-684 0d 684-686 0d 686-688 0d 688-690 0d 690-692 0d 692-694 0d 694-696 0d 696-698 0d 698-700 0d 700-702 0d 702-704 0d 704-706 0d 706-708 0d 708-710 0d 710-712 0d 712-714 0d 714-716 0d 716-718 0d 718-720 0d 720-722 0d 722-724 0							

1. LOCATION OF WELL		WATER WELL RECORD		2. PROPERTY OWNER'S NAME	
County Name		Minnesota Signature		Date of Completion	
Township Name	Township Number	Range Number	Section No.	Address	
Distance and Direction from Road Intersection or Street Address and City of Well Location				St. Louis Park Well #3	
				2924 Idaho Ave.	

NON-RESPONSIVE

DRIFT
PLATEVILLE LIMESTONE
ST. PETER SAND
RED SHALE
SHALE

0 103
03 118
118 230
230 245
245 286

4. WELL IDENTIFICATION (Completed)		Date of Completion	
286		August, 1959	
5. WELL TYPE			
<input type="checkbox"/> Casing <input type="checkbox"/> Drift <input type="checkbox"/> Gravel <input type="checkbox"/> Sand <input type="checkbox"/> Silt <input type="checkbox"/> Other			
6. USE			
<input type="checkbox"/> Domestic <input type="checkbox"/> Irrigation <input type="checkbox"/> Test Well <input type="checkbox"/> Public Supply <input checked="" type="checkbox"/> Municipal <input type="checkbox"/> Air Conditioning <input type="checkbox"/> Industry <input type="checkbox"/> Commercial			
7. CASING		HEIGHT: Above/Below	
<input type="checkbox"/> Black <input type="checkbox"/> Galv. <input type="checkbox"/> Other		<input type="checkbox"/> Threaded <input type="checkbox"/> Welded <input type="checkbox"/> Other	
In. to 103 ft. Weight lbs./ft. In. to ft. Weight lbs./ft. In. to ft. Weight lbs./ft.		Drive Shaft Yes No Hole Diam. In. to ft. In. to ft. In. to ft.	
8. MAKE			
Type _____ Dia. _____			
Slot/Gauge _____ Length _____			
Set between _____ ft. and _____ ft.			
_____ ft. and _____ ft.			
_____ ft. and _____ ft.			
9. STATIC WATER LEVEL			
50 ft. below land surface			
10. PUMPING LEVEL (Below land surface)			
164 ft. after hrs. pumping 1350			
ft. after hrs. pumping			
11. WELL HEAD COMPLETION			
<input type="checkbox"/> Pressure Adapter <input type="checkbox"/> Flowmeter offset <input type="checkbox"/> At least 12" above grade			
12. Well grouted?			
<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Heat Cement <input type="checkbox"/> Bentonite <input type="checkbox"/> Other			
Depth from _____ ft. to _____ ft.			
from _____ ft. to _____ ft.			
13. Nearest sources of possible contamination			
_____ feet _____ direction _____ type			
Well disinfected upon completion? Yes <input type="checkbox"/> No <input type="checkbox"/>			
14. PUMP			
Date installed _____			
<input type="checkbox"/> Not installed			
Manufacturer's Name _____			
Model Number _____ HP _____ Volts _____			
Length of drop pipe _____ ft. capacity _____ g.p.m.			
Material of drop pipe _____			
Type: <input type="checkbox"/> Submersible <input type="checkbox"/> Jet <input type="checkbox"/> L.S. Turbine <input type="checkbox"/> Centrifugal <input type="checkbox"/> Other			
15. WATER WELL CONTRACTOR'S CERTIFICATION			
This well was drilled under my production and this report is true to the best of my knowledge and belief.			
McCarthy Well Co.			
License No. _____			
Address _____			
Signed _____ Date _____			
Authorized Representative			
Name of Driller _____ Date _____			

15. REMARKS, ELEVATION, SOURCE OF DATA, etc.

Elevation = 214.2

REPORT

Tack No.

Well No. 3

Town St. Louis Park

Date Started Machine No. State Minnesota

Date Completed August 1939 Owner Village

Location by the Tower Total Depth of Well

DIAMETER OF HOLE	24"				
Top of Pipe ^{above} Surface					
Bottom of Pipe below Surface	103'				
No. of Ft. of Pipe in the Hole					
No. of Ft. of Hole Drilled					

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole	286				Drift	103	103
Depth to Water at Rest	72				Limerock	15	118
Depth to Water Pumping					Sandrock	112	230
Depth of Pump Pipe					Red Shale	15	245
Size of Cylinder					Shale & Sandrock	41	286
Length of Stroke							
Strokes per minute							
Gallons per minute							
Will well supply more?					12/15/51-From Vralsted's eco ds.		
Was Strainer in Hole?					12/31/51-Static 44' with pump on		
Was water clear?					Wells #1 & #2 running.		
Was well pumping sand?					Static level 63' with #3		
Hours putting in Pump					only running. Water level 85		
Hours Pumping							
Hours taking out Pump							
Hours Consumed							

STRAINER

Make		
Type of Metal		
Diameter O. D.		
Diameter I. D.		
Total Length		
Number		
Top of Screen below Surface		
No. of Ft. Exposed		
Bottom of Screen below Surface		
Was Str. swedged		
Did Sand come thru Str.		
Was Str. coarse enough		
Style of Fittings		

All measurements taken from

LAYNE-WESTERN COMPANY

OF MINNESOTA

FIELD REPORT OF COMPLETED WELL

Name of Job St. Louis Park, Minn. Date started 4/17/46
 Address No. 4 Date completed _____
NON-RESPONSIVE No. of days _____

LOG OF WELL

From	To	Material	From	To	Material
0	3	Dirt fill	291	355	Hard lime
3	76	Sand & gravel	355	398	Lime, milky cuttings
76	106	Platteville Lime.	398	445	Jordan sandstone
106	235	Soft St. Peter Sand.	445	455	Jordan Sandstone & shale, ^{hard}
235	277	Hard sandstone & shale	455	470	" "
277	291	Hard lime	470	490	St. Lawrence

Kind of plug in well none Static water level _____
 Depth of well—ground level to top of plug _____

MATERIAL LEFT IN WELL

	Opening	Length - feet	Diameter - inches	Material
Screen				
Inner casing		304'	18" O.D.	
Outer casing		89'10"	24" O.D.	

Was outer casing cemented _____ Amount _____
 Size of gravel used _____ in. to _____ in. Amount _____

TEST OF WELL

Hours Pumped		Yield gal. per min.	Water Level ft. below surface	Remarks
From	To			
9/18 4PM	9/19 4 PM	2500	82'	drawdown averages 53' length of airline 180' to top of bowls

Did well clear up yes Time to clear approx. 4 hours
 Date _____ Driller _____

REPORT

Tack No.

Well No. 4

Town St. Louis Park

Date Started Machine No. State Minnesota

Date Completed 4/7/46 Owner Village

Location NON-RESPONSIVE Total Depth of Well

DIAMETER OF HOLE	24"	18"			
Top of Pipe ^{above} Surface.....					
Bottom of Pipe below Surface.....	89' 10"	304'			
No. of Ft. of Pipe in the Hole.....					
No. of Ft. of Hole Drilled.....					

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole.....	490				Dirt Fill	0	3
Depth to Water at Rest.....	72				Sand & Gravel	73	76
Depth to Water Pumping.....	125				Platteville	30	106
Depth of Pump Pipe.....					St. Peter soft.	129	235
Size of Cylinder.....					Hard Sandstone & Shale	42	277
Length of Stroke.....					Hard Lime	73	355
Strokes per minute.....					Lime Milky Cuttings	43	398
Gallons per minute.....	2500				Jordan Sandstone	47	445
Will well supply more?.....					Jordan & Shale hard.	10	455
Was Strainer in Hole?.....					Jordan Sandstone	15	470
Was water clear?.....					St. Lawrence	20	490
Was well pumping sand?.....							
Hours putting in Pump.....							
Hours Pumping.....							
Hours taking out Pump.....							
Hours Consumed.....							

12/15/51-Above from Vralsted's Record
12/31/51-Static 63'.

STRAINER

Make.....		
Type of Metal.....		
Diameter O. D.....		
Diameter I. D.....		
Total Length.....		
Number.....		
Top of Screen below Surface.....		
No. of Ft. Exposed.....		
Bottom of Screen below Surface.....		
Was Str. swedged.....		
Did Sand come thru Str.....		
Was Str. coarse enough.....		
Style of Fittings.....		

All measurements taken from

REPORT

Tack No.

Well No. 5

Town St. Louis Park

Date Started 5/28/47

Machine No. State Minnesota

Date Completed 8/21/47

Owner Village

Location NON-RESPONSIVE

Total Depth of Well

DIAMETER OF HOLE	24"	20"			
Top of Pipe ^{above} Surface					
Bottom of Pipe below Surface	115'	305'			
No. of Ft. of Pipe in the Hole					
No. of Ft. of Hole Drilled					

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole	465				Sand & Gravel	5	5
Depth to Water at Rest	87				Clay & Boulders	10	15
Depth to Water Pumping					Sand & Gravel	88	103
Depth of Pump Pipe					Coarse Gravel	6	109
Size of Cylinder					Lime	11	120
Length of Stroke					Rock & Shale	12	132
Strokes per minute					St. Peter	98	230
Gallons per minute					Shale & Rock	55	285
Will well supply more?					Shakopee Lime	122	407
Was Strainer in Hole?					Jordan Sand	53	460
Was water clear?					St. Lawrence	5	465
Was well pumping sand?							
Hours putting in Pump							
Hours Pumping							
Hours taking out Pump							
Hours Consumed							

15 Yds. cement used.

12/15/51-Above from Vral. ted's Reco

12/31/51-Static 76' at 1'00 GPM

Water drew down to 88'.

STRAINER

Make		
Type of Metal		
Diameter O. D.		
Diameter I. D.		
Total Length		
Number		
Top of Screen below Surface		
No. of Ft. Exposed		
Bottom of Screen below Surface		
Was Str. swedged		
Did Sand come thru Str.		
Was Str. coarse enough		
Style of Fittings		

All measurements taken from

LAYNE-WESTERN COMPANY

OF MINNESOTA

FIELD REPORT OF COMPLETED WELL

Name of Job St. Louis Park, Minnesota Date started 9/30/47
 Address 2.6 Date completed 1/19/48
 No. of days 73

LOG OF WELL

From	To	Material	From	To	Material
0	90	sand & gravel	480	482	St. Lawrence
90	122	Platteville lime			
122	127	Blue shale			
127	290	St. Peter sand - soft			
290	417	Shakopee Lime			
417	480	Jordan sandstone			

Kind of plug in well none Static water level 60'
 Depth of well-ground level to top of plug 482'

MATERIAL LEFT IN WELL

	Opening	Length - feet	Diameter - inches	Material
Screen				
Inner casing		303'	20" O.D.	welded
Outer casing		107'6"	24" O.D.	welded

Was outer casing cemented yes Amount 24 yards
 Size of gravel used _____ in. to _____ in. Amount _____

TEST OF WELL

Hours Pumped		Yield	Water Level	Remarks
From	To	gal. per min.	ft. below surface	
Test of well		appears on another report		

Did well clear up _____ Time to clear _____
 Date 1/19/48 Driller Paul W. Shuey

REPORT

Tack No.

Well No. 6

Town St. Louis Park

Date Started 9/20/47

Machine No. State Minn.

Date Completed 1/19/48

Owner Village

Location NON-RESPONSIVE

Total Depth of Well

DIAMETER OF HOLE	24"	20"			
Top of Pipe ^{above} Surface					
Bottom of Pipe below Surface	107' 6"	303'			
No. of Ft. of Pipe in the Hole					
No. of Ft. of Hole Drilled					

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole	482				Sand & Gravel	90	90
Depth to Water at Rest	60				Platteville	32	122
Depth to Water Pumping					Blue Shale	5	127
Depth of Pump Pipe					St. Peter soft.	163	290
Size of Cylinder					Shakopee Lime	127	417
Length of Stroke					Jorden Sandstone	63	480
Strokes per minute					St. Lawrence	2	482
Gallons per minute							
Will well supply more?							
Was Strainer in Hole?					24 Yds. cement used.		
Was water clear?							
Was well pumping sand?					12/15/51-Above from Vraal ted'		Recor
Hours putting in Pump					12/31/51-Static 55'. At 1 00 G M		
Hours Pumping					water drew down o 81 .		
Hours taking out Pump							
Hours Consumed							

STRAINER

Make		
Type of Metal		
Diameter O. D.		
Diameter I. D.		
Total Length		
Number		
Top of Screen below Surface		
No. of Ft. Exposed		
Bottom of Screen below Surface		
Was Str. swedged		
Did Sand come thru Str.		
Was Str. coarse enough		
Style of Fittings		

All measurements taken from

LAYNE - MINNESOTA CO.

FLOR 152. 192.20

FIELD REPORT OF COMPLETED WELL

Name of job St. Louis Park Date started 3/10/52
 Address NON-RESPONSIVE Date completed 5/9/52
No 7 No. of days 41

LOG OF WELL

From	To	Material	From	To	Material
0	75	Sand gravel & Boulders	380	420	Good Gordon Sand.
75	97	Flatville Lime	420	430	Fine Sand & Shale
97	100	Shale	430	440	Coarse Jordan Sand
100	210	Saint Peter Sand.	440	446	St. Lawrence.
210	260	Shale & Sand			
260	380	Dolemite lime.			

Kind of plug in well none Static water level 58
 Depth of well - ground level to top of plug 446

MATERIAL LEFT IN WELL

Opening	Length - feet	Diameter - inches	Material
Screen <u>none</u>			
Inner casing	<u>274</u>	<u>20"</u>	<u>Welded</u>
Outer casing	<u>80</u>	<u>24"</u>	<u>Welded</u>

Was outer casing cemented Drove Amount

Size of gravel used in. to in. Amount

Inner Casing Cemented 25 sacks plus 10 yds. Ready Mix

TEST OF WELL

Hours Pumped		Yield	Water Level	Remarks
From	To	gal. per min.	ft. below surface	

Did well clear up Time to clear

Date 5/12/52 Driller Paul W. Shuey

REPORT

Tack No.

Well No. 7

.....Town St. Louis Park

Date Started.....Machine No.....State Minnesota

Date Completed.....Owner City

NON-RESPONSIVE

Total Depth of Well.....446'

DIAMETER OF HOLE	24"OD	20"OD			
Top of Pipe ^{above} Surface					
Bottom of Pipe below Surface	80'	247'			
No. of Ft. of Pipe in the Hole					
No. of Ft. of Hole Drilled					

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole.....	51				Drift	75	75
Depth to Water at Rest.....	58				Plattville Limerock	25	100
Depth to Water Pumping.....					St. Peter Sandrock	160	260
Depth of Pump Pipe.....					Shakopee	120	380
Gallons per minute.....					Jordan Sandrock	60	440
Will well supply more?.....					St. Laurence	6	446
Was Strainer in Hole?.....							
Was water clear?.....					25 sacks of cement		
Was well pumping sand?.....					10 yds. of ready mix		
Hours Pumping.....							

STRAINER

Make.....		
Type of Metal.....		
Diameter O. D.....		
Diameter I. D.....		
Total Length.....		
Number.....		
Top of Screen below Surface.....		
No. of Ft. Exposed.....		
Bottom of Screen below Surface.....		
Was Str. swedged.....		
Did Sand come thru Str.....		
Was Str. coarse enough.....		
Style of Fittings.....		

~~XXXX~~

Above information from
Layne report and city.

All measurements taken from

REPORT

Tack No.

Town St Louis Park

Date Started 3/10/52

Machine No. State MINN

Date Completed 5/9/52

Owner Village

Location 2500 Louisiana Av

Total Depth of Well 446

DIAMETER OF HOLE	24"	20"			
Top of Pipe ^{above} below Surface					
Bottom of Pipe below Surface					
No. of Ft. of Pipe in the Hole	<u>80'</u>	<u>274'</u>			
No. of Ft. of Hole Drilled		<u>446</u>			

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole					<u>Drift</u>	<u>75</u>	<u>75</u>
Depth to Water at Rest	<u>58</u>				<u>Platteville</u>	<u>22</u>	<u>97</u>
Depth to Water Pumping					<u>Shale</u>	<u>3</u>	<u>100</u>
Depth of Pump Pipe					<u>St Peter</u>	<u>110</u>	<u>210</u>
Size of Cylinder					<u>Shale + Sand</u>	<u>50</u>	<u>260</u>
Length of Stroke					<u>Dalomite - Shakopee</u>	<u>120</u>	<u>380</u>
Strokes per minute					<u>Jordan</u>	<u>40</u>	<u>420</u>
Gallons per minute					<u>Fine Sand & Shale</u>	<u>10</u>	<u>430</u>
Will well supply more?					<u>Coarse - Jordan</u>	<u>10</u>	<u>440</u>
Was Strainer in Hole?					<u>Shale</u>	<u>6</u>	<u>446</u>
Was water clear?							
Was well pumping sand?							
Hours putting in Pump							
Hours Pumping							
Hours taking out Pump							
Hours Consumed							

STRAINER

Make		
Type of Metal		
Diameter O. D.		
Diameter I. D.		
Total Length		
Number		
Top of Screen below Surface		
No. of Ft. Exposed		
Bottom of Screen below Surface		
Was Str. swedged		
Did Sand come thru Str.		
Was Str. coarse enough		
Style of Fittings		

El Floor of Pump R
194 + 710.3 = 904.3

All measurements taken from

WELL #8 - CITY OF ST. LOUIS PARK, MINN.

Built
1956

DRIFT

STATIC WATER
LEVEL - 95' - 10 1/2'

102'

253'-1"
24" O.D. CASING

177' SAND ROCK

CEMENT
GROUT

314'-2"
16" O.D. CASING

307'

BRIDGE

110' LIMEROCK

192'-10"

23'
HOLE

62' SAND ROCK

BERGERSON-CASELL
A R HART 11/1/55

SHALEY SAND ROCK

REPORT

Tack No.

Well No. 8

G. B. C. Inc

Town St. Louis Park

Date Started 6/10/55

Machine No. State Minnesota

Date Completed 11/2/55

Owner City

NON-RESPONSIVE

Total Depth of Well 507'

DIAMETER OF HOLE	24" O.D.	16" O.D.			
Top of Pipe ^{above} Surface	3'	3'1"			
Bottom of Pipe below Surface	250'1"	311'1"			
No. of Ft. of Pipe in the Hole	253'1"	314'2"			
No. of Ft. of Hole Drilled	507'				

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole	507	507	507		Platform	3	3
Depth to Water at Rest					Yellow Clay, Rocks & Boulders	35	38
Depth to Water Pumping	143	115	111		Hard red clay & rocks	27	65
Depth of Pump Pipe					Brown gray clay & rocks	32	97
Gallons per minute	1750	1000	800		Sand & gravel (dirty)	11	108
Will well supply more?					Clay, rocks & broken limerock - Gravel & broken limerock-Platteville	18	126
Was Strainer in Hole?					St. Peter formation	177	303
Was water clear?					Yellow shale	6'	
Was well pumping sand?					Sandrock & shale	47	
Hours Pumping					Sandrock	63	
					Shale	17	
					Sandrock (Hard)	43	
					Shale & Sandrock	1	
					Shakopee limerock (Hard)	110	413
					Jordan formation:	82	495
					Sandrock-Med. hard & clean	23'	
					" Fine, hard, red	28	
					" Very hard, shaly	18	
					" Med. hard, white, coarse	4	
					" Med. hard, white, fine	10	
					Shaly Sandrock	12	507'

STRAINER

Make		
Type of Metal		
Diameter O. D.		
Diameter I. D.		
Total Length		
Number		
Top of Screen below Surface		
No. of Ft. Exposed		
Bottom of Screen below Surface		
Was Str. swedged		
Did Sand come thru Str.		
Was Str. coarse enough		
Style of Fittings		

All measurements taken from Grade
EL 931.3'

10/25/55 Ira Vraalstad reports sand
content of water @ 1000 GPM -13 PPM
800 GPM -0.6 PPM

LAYNE - MINNESOTA CO.

FIELD REPORT OF COMPLETED WELL

Name of Job St. Louis Park, Minnesota Date started _____
 Address Well # 9 Date completed 6-6-56
 _____ No. of days _____

LOG OF WELL

From	To	Material	From	To	Material
0	69	Drift	345	380	Shakopee Limestone
69	120	Platteville Limestone	380	473	Jordan Sandstone
120	220	Shale and sandstone			
220	275	Shale			
275	339	Shakopee Limestone			
339	345	Red Sandstone			

Kind of plug in well Pressure Grouted Liner Static water level 70'
 Depth of well-ground level to top of plug _____

MATERIAL LEFT IN WELL

	Opening	Length - feet	Diameter - inches	Material
Screen				
Inner casing		289'	16"	Steel
Outer casing		81'	24"	Steel

Was outer casing cemented Yes Amount 2414 Bags
 Size of gravel used _____ in. to _____ in. Amount _____

TEST OF WELL

Hours Pumped		Yield gal. per min.	Water Level ft. below surface	Remarks
From	To			
				See Pump test report

Did well clear up _____ Time to clear _____

Date 6-8-56 Driller Wayne La Fontaine

E. H. Renner & Sons

WELL DRILLING FOR FOUR GENERATIONS
6300 Industry Ave. N. W., Anoka, Minn. 55303, (612) 427-6100

WELL LOG #10

MAP CODE _____

Date Started _____ 19 **55**

Date Completed _____ 19 _____

Owner or Contractor **City of St. Louis Park**

Address **NON-RESPONSIVE**

Job Location **29th and Idaho area**

Lot _____ Block _____ City **St. Louis Park** County **Hennepin** State of Minnesota

Well: ☒ Cable Tool ☒ Rotary ☒ Driven Driller **Kay's Well Drilling Co.**

Cased with **24** inch ☒ PE ☒ T&C **315'-6"** Ft. Total Depth of Well **500** Ft. from grade

Feet of Open Hole **185** Finished in **Jordan Sandstone** Static Water Level **104** Ft.

Tested at **2055** gallons per min. Drawn down of **100** feet.
1060

Screen: Size _____ dia. _____ ft. Make _____ Slot or Gauge _____

Pump: Make _____ HP. _____ Volts _____ Phase _____ Type _____ Tank _____

Motor Serial No. _____ Pump Serial No. _____ Drop Pipe _____ feet

Size _____ Capacity of pump _____ G.P.M. Date Installed _____

Pitless Adapter: Make _____ Offset _____ ft. Material _____ Size _____ inch

Kind of Formation	Color of Formation	Started Depth	Ended Depth	Total Thickness of Formation	Remarks
Sand & Gravel		0	85	85	
Clay		85	103	18	Well is grouted from 315' to surface 1015 bags cement
Platville		103	125	22	
St. Peter		125	290	165	
Shakopee Dolomite		290	409	119	
Jordan		409	500	91	

REPORT

Tack No.

Well No. 10

Town St. Louis Park

Date Started Machine No. State Minnesota

Date Completed Owner City of St. Louis Park

Location Total Depth of Well

DIAMETER OF HOLE	24"	16"		
Top of Pipe ^{above} Surface.....				
Bottom of Pipe below Surface.....	106	315		
No. of Ft. of Pipe in the Hole.....				
No. of Ft. of Hole Drilled.....				

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole.....					Drift		108
Depth to Water at Rest.....	04				Platteville		125
Depth to Water Pumping.....	200				St. Peter		290
Depth of Pump Pipe.....					Shakopee/Oneota		290
Gallons per minute.....	00				Jordan		409
Will well supply more?.....					St. Lawrence		500
Was Strainer in Hole?.....							
Was water clear?.....							
Was well pumping sand?.....							
Hours Pumping.....							

STRAINER

Make.....		
Type of Metal.....		
Diameter O. D.....		
Diameter I. D.....		
Total Length.....		
Number.....		
Top of Screen below Surface.....		
No. of Ft. Exposed.....		
Bottom of Screen below Surface.....		
Was Str. swedged.....		
Did Sand come thru Str.....		
Was Str. coarse enough.....		
Style of Fittings.....		

All measurements taken from

REPORT

Tack No. 258 Green
 Well No. 11
 By Bergerson-Caswell Inc. Town St. Louis Park
 Date Started 6-13-60 Machine No. _____ State Minnesota
 Date Completed 11-1-60 Owner City of St. Louis Park
 Location NON-RESPONSIVE Total Depth of Well 1093'

DIAMETER OF HOLE	24"	16"			
Top of Pipe above Surface	1	-9'8"			
Bottom of Pipe below Surface	102	880			
No. of Ft. of Pipe in the Hole	103	870'4"			
No. of Ft. of Hole Drilled	777	213'			

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole	1093		1093		Drift	101	101
Depth to Water at Rest	221	221	221	221	lattville Limerock	19	120
Depth to Water Pumping	321	330	329	324	St. Peter Sandrock	168	288
Depth of Pump Pipe					Shakopee-Oneota limerock	120	408
Gallons per minute	1200	1150	1200	1500	Jordan sandrock	97	505
Will well supply more?	Yes	Yes	Yes	Yes	St. Laurence-limerock-shal	25	530
Was Strainer in Hole?	Yes	Yes	Yes	Yes	Franconia-hard, sticky		
Was water clear?	No	No	No	No	green shale	153	683
Was well pumping sand?	Yes	Yes	Yes	Yes	Dresbach	272	955
Hours Pumping	10 1/2	10 1/2	10 1/2	10 1/2	Gailesville-clean hard SR		

STRAINER

Make			
Type of Metal			
Diameter O. D.			
Diameter I. D.			
Total Length			
Number			
Top of Screen below Surface			
No. of Ft. Exposed			
Bottom of Screen below Surface			
Was Str. swedged			
Did Sand come thru Str.			
Was Str. coarse enough			
Style of Fittings			

& shale 683' to 745' (62')
 Eau Claire-hard grey shale
 745' to 805' (60')
 yellow shale & sandrock
 805' to 813' (8')
 green shale
 813 to 817 (4')
 Sandrock & shale
 817 to 853 (36')
 Mt. Simon
 hard sandrock and shale
 853 to 955 (102')
 Hinckley 123 1078
 Pinkish red sandrock som
 shale 955' to 1050' (95')
 Hard clean coarse S.R.
 1050' to 1078' (28')
 Red Clastic 15 1093'

All measurements taken from platform-
 approximately 12" above perm.
 grade.

SHOT WELL #1--50# @ 1070'
 #2--50# @ 1060'
 #3--50# @ 1050'
 #4--50# @ 1030'
 #5--50# @ 1010'

Calculated on number of bailers removed
 and bailer fill--est. removed 225 1/2 Cu. Yds.
 182 after shooting & bailing & testing pumping
 pulled pump and found hole filled to 1024'
 384 Bailed clean and reinstalled test pump.

12/5/60 Mailed Minn. Consv. Dept. Well log sheet jwm

WELL RECORD

KEYS WELL DRILLING COMPANY WATER PRODUCERS SAINT PAUL, MINNESOTA

Owner ST. LOUIS PARK Date Completed August, 1963
Location NON-RESPONSIVE Driller Kemper
Well No. 18 Size 30x24x16 Total Depth 1095 Type _____

DRILLERS LOG

0 ' to 96 ' Drift
96 ' to 127 ' Platville
127 ' to 132 ' Shale (Glenwood)
132 ' to 292 ' St. Peter
292 ' to 427 ' Shakopee
427 ' to 505 ' Jordan
505 ' to 550 ' St. Lawrence
550 ' to 695 ' Franconia
695 ' to 725 ' Ironton
725 ' to 745 ' Dressbach (Galena)
745 ' to 832 ' Dressbach (Eau Claire)
832 ' to 983 ' Dressbach (Mt. Simon)
983 ' to 1095 ' Hinckley
_____ ' to _____ ' _____

PERMANENT PUMP DATA

Mfg. _____ Type _____ Serial No. _____
Capacity _____ GPM _____ TDH _____
Motor Make _____ Type _____
_____ H. P. _____ Volts _____ Ph. _____ RPM _____
_____ ft. _____ in Col. pipe _____ in. Shaft _____
_____ ft. _____ in Bore _____ Stages _____ Type _____
_____ ft. _____ in suction pipe & _____
_____ ft. Total Length of Pump _____
_____ ft. _____ in. drop pipe & _____ No. Cable _____
_____ ft. _____ in. air line _____
_____ in. Pitless _____ ft. bury _____ in outlet _____

WELL MATERIALS

99 ' of 30 " diameter of Outer Casing
160 ' of 29 " diameter of Open Hole
270 ' of 24 " diameter of Inner Casing
825 ' of 23 " diameter of Open Hole
900 ' of 16 " diameter of Open Hole
_____ ' to _____ Mix grout 1860 (Sacks)
_____ " diameter _____ Screen

RECORD OF TEST PUMPING

Static Water Level 245 ft. from _____
863 GPM 86 D.D. 11 Hours
1012 GPM 95 D.D. 8 Hours
1235 GPM 117 D.D. 8 Hours
1218 GPM 114 D.D. 11 Hours
1455 GPM 127 D.D. 18 Hours
1585 GPM 128 D.D. 4 Hours
1711 GPM 143 D.D. 4 Hours
1795 GPM 154 D.D. 5 Hours
_____ GPM _____ D.D. _____ Hours

Remarks: 580' of Hinckley sandrock removed
after blasting & bailing.

1863 GPM 157' D.D. 11 Hours
2000 156' 2
1500 122'6" 13
1300 108' 1
1100 93' 1

LAYNE-MINNESOTA CO.

FIELD REPORT OF COMPLETED WELL

Name of Job St. Louis Park Well No. 13 Date started Oct. 1, 1963
Address NON RESPONSIVE Date completed July 1, 1964
Minneapolis, Minnesota No. of days _____

LOG OF WELL

From	To	Material	From	To	Material
386'	460'	Gordon Sand	770'	778'	Mt. Simon Sandstone
460'	490'	St. Lawrence	778'	917'	Hit layers of 1' to 2' thick shale in sand formation
490'	655'	Franconia			
655'	714'	Dresbach	917'	1040'	Hinkley Sandstone
714'	770'	Hard rubber shale	1040	1045	Red Clastic Formation filled Lt. back in again

Method of drilling _____ Rig used _____ Diam. of drill hole _____ in.
Was outer casing cemented _____ Method _____ Amount cement _____
Depth of well - from ground level _____ ft. - from top of casing _____ ft. Static _____ ft.

MATERIAL LEFT IN WELL

	Opening	Length - feet	Diameter - inches	Material
Screen				
Inner casing				
Outer casing				

Under reamed from _____ ft. to _____ ft. Diameter _____ in. Method _____
Size-sizes-of gravel 1/4 by 1/2 Rock Backfill Amount 33 yards
Well shot at _____ ft. to _____ ft. depth Number _____ Size _____ lb. Material removed _____ yd.

WELL TEST

Hours Pumped		Yield gal. per min.	Water Level- ft. below surface	Drawdown feet	Remarks
From	To				

Time to clear _____ Specific Capacity _____ g.p.m./ft. d.d.
Date July 1, 1964 Driller Gordon Hollen

REPORT

Tack No.

Well No. 14

Town St. Louis Park

Date Started 8/27/64

Machine No. State Minn.

Date Completed

Owner City of St. Louis Park

NON-RESPONSIVE

Total Depth of Well 485'

DIAMETER OF HOLE	30"	24"	16"		
Top of Pipe ^{above} Surface	-10'	0	0	press. gr	outed betwe
Bottom of Pipe below Surface	94'	253'	389'	164'	24' & 24' &
No. of Ft. of Pipe in the Hole	84'				30" (710 sa
No. of Ft. of Hole Drilled	159'	137'	96'		

TEST	1	2	3	4
Depth of the Hole	5	485	485	48
Depth to Water at Rest	80	80	80	8
Depth to Water Pumping	08	113	119	125
Depth of Pump Pipe				
Gallons per minute	600	700	800	100
Will well supply more?	yes	yes	yes	ye
Was Strainer in Hole?	no	no	no	no
Was water clear?	s	yes	yes	ye
Was well pumping sand?				
Hours Pumping PPM	.0	5.0	9.0	12.

STRAINER

Make			
Type of Metal			
Diameter O. D.			
Diameter I. D.			
Total Length			
Number			
Top of Screen below Surface			
No. of Ft. Exposed			
Bottom of Screen below Surface			
Was Str. swedged			
Did Sand come thru Str.			
Was Str. coarse enough			
Style of Fittings			

FORMATION	Thickness	Depth
Platform	3	3
Glacial drift	91	94
Plattville limerock	4	98
Glenwood shale	3	101
St. Peter sandrock	164	265
Shakopee/Oneota limeroc	10	275
Jordan sandrock	110	85
Hard-med., clean-coarse		
375-410		
Very hard - red.		
410-420		
Hard shaley		
420-440		
Hard-fine-tan-poor		
440-450		
Hard-fine, white, tan, poc		
450-475		
Hard, green shaly, fine		
475-485		

Jordan sandrock was sho -
removed 147 cu. yds. sa roc .

All measurements taken from grade plus 3'

El. 905' plus or minus

Tack No. _____
 Well No. 15
 Town St. Louis Park
 Date Started 5/22/69 Machine No. _____
 Date Completed 10/24/69 Owner City of St. Louis Park
 Location _____ Total Depth of Well _____

DIAMETER OF HOLE	30"	24"
Top of Pipe ^{above} Surface		
Bottom of Pipe below Surface	102	398
No. of Ft. of Pipe in the Hole		
No. of Ft. of Hole Drilled	297	105

TEST	1	2	3	4	FORMATION	Water	De
Depth of the Hole					Glacial drift	102	102
Depth to Water at					Platthub limestone	22	124
Depth to Water Pump					St. Peter sandrock	64	288
Depth of Pump Pipe					Shakopee/Onota limestone	114	402
Gallons per minute					Jordan sandrock	80	482
Will well supply more?					St. Lawrence shale	21	503
Was Strainer in Hole?							
Was water clear?							
Was well pumping sand?							
Hours Pumping							

STRAINER

Make	
Type of Metal	
Diameter O. D.	
Diameter I. D.	
Total Length	
Number	
Top of Screen below Surface	
No. of Ft. Exposed	
Bottom of Screen below Surface	
Was Str. swaged	
Did Sand come thru Str.	
Was Str. coarse enough	
Style of Fittings	

Well shot & bailed
 removed 182 cu. yds.

All measurements taken from Trade

TRI-STATE DRILLING CO.

Owner ST. LOUIS PARK
NON-RESPONSIVE
 Location

Well Designation Deep Well No. 16

Total Depth 500 feet.

DRILLER'S LOG

<u>0</u>	to	<u>60</u>	<u>sand & gravel</u>
<u>60</u>	to	<u>80</u>	<u>clay & bolders</u>
<u>80</u>	to	<u>105</u>	<u>coarse sand & gravel</u>
<u>105</u>	to	<u>118</u>	<u>broken limestone</u>
<u>118</u>	to	<u>128</u>	<u>platteville limestone</u>
<u>128</u>	to	<u>258</u>	<u>St. Peter sandstone</u>
<u>258</u>	to	<u>294</u>	<u>red shale</u>
<u>294</u>	to	<u>410</u>	<u>Shakopee limestone</u>
<u>410</u>	to	<u>495</u>	<u>Jordan sandstone</u>
<u>495</u>	to	<u>500</u>	<u>St. Laurance shale</u>
<u> </u>	to	<u> </u>	<u> </u>
<u> </u>	to	<u> </u>	<u> </u>
<u> </u>	to	<u> </u>	<u> </u>
<u> </u>	to	<u> </u>	<u> </u>
<u> </u>	to	<u> </u>	<u> </u>
<u> </u>	to	<u> </u>	<u> </u>
<u> </u>	to	<u> </u>	<u> </u>

TEST PUMPING DATA

Static water level 125 feet. Pumped at 2,000 g.p.m.

with 238 foot level 2 P.P.M. sand at

1200 G.P.M.

Date completed 7/31/73

Driller Frank Berthiaume

Well Type: ☒ Rock

☐ Screen

☐ Gravel Packed

WELL MATERIALS

410 ft. 30 in. diam. outer casing

425 ft. 24 in. diam. liner pipe

 ft. in. diam. screen

Screen type

Remarks: Well liner grouted in.

Jordan sandstone was developed by

blasting & air surging & bailing.

PERMANENT PUMP DATA

Mfg. Model

Serial No. Type

 h.p. Motor, V. Ph.

 ft. setting in. shaft in. col. pipe

Remarks:

DEPARTMENT OF HEALTH
WATER WELL RECORD
Minnesota Statute 136A.01, 02

MINNESOTA UNIQUE WELL NO.
per Water Sample

147459

St. Louis Park

34th & L Ing - Well #17

City of St. Louis Park
St. Louis Park, MN

NON-RESPONSIVE

FORMATION LOG	COLOR	THICK.	FROM	TO
Drift			0	105'
Drift w/ of Lime k			05'	115'
Shale	Blue		15'	124'
St. Peter Sandstone			24'	227'
Shale	Red		7.	275'
Shakopee Limestone			75.	405'
Jordan Sandstone			05'	465'
Shale	Red & G		5'	691'
Sandstone w/Shale Lay			91'	805'
Sandstone			5'	1082'
Red Clastics			1082	1085

1. WELL IDENTIFICATION (continued)

2. WELL USE

3. WELL CONSTRUCTION

4. WELL EQUIPMENT

5. WELL PROTECTION

6. WELL RECORD

7. WELL WATER LEVEL

8. PUMPING LEVEL (below land surface)

9. WELL TEST

10. WELL GROUTING

11. NEAREST SOURCES OF POSSIBLE CONTAMINATION

12. WELL DISINFECTED UPON COMPLETION?

13. PUMP

14. WATER WELL CONTRACTOR'S CERTIFICATION

18" casing to 475'
12" casing to 818'
12" open hole to 1,085'

ALL CASINGS ARE .375
WALL THICKNESS

Layne Minnesota Company 101
3147 California St. NE, Wpls.

5-25-93

25-83

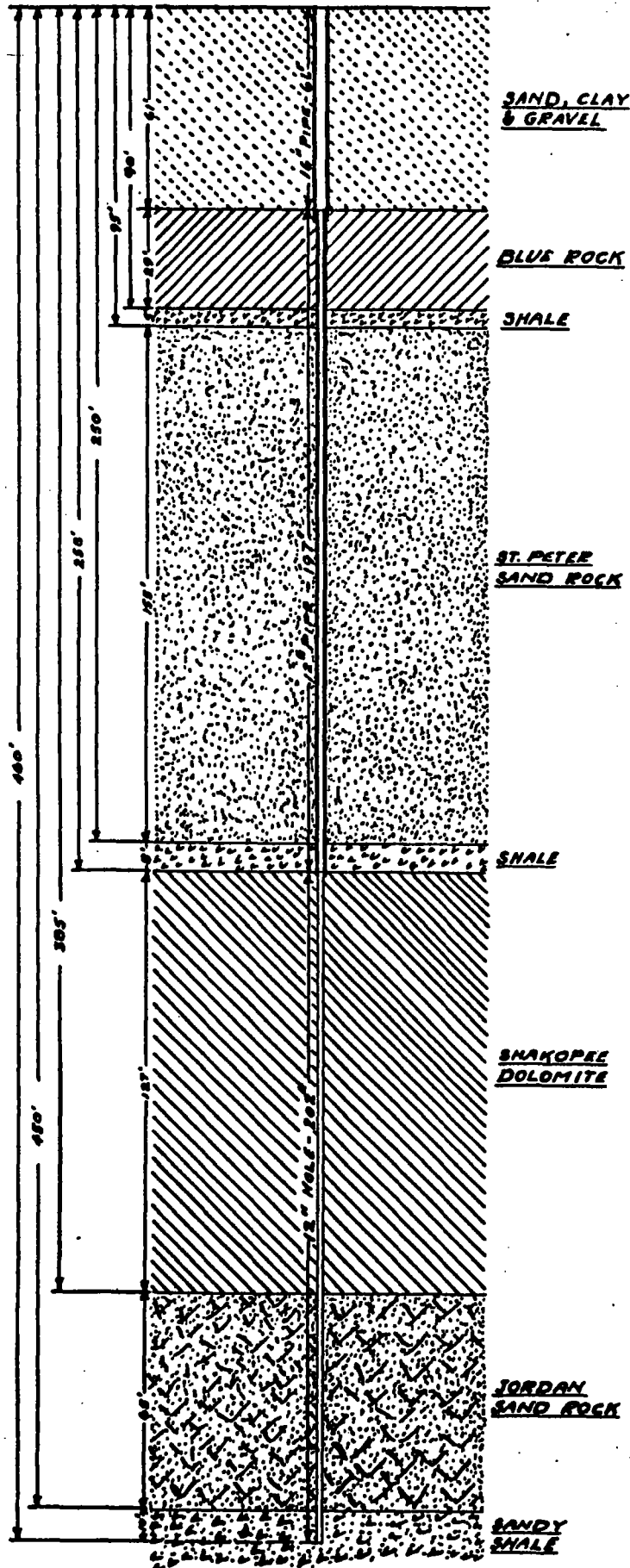
COUNTRY CLUB DISTRICT SERVICE CORPORATION WELL

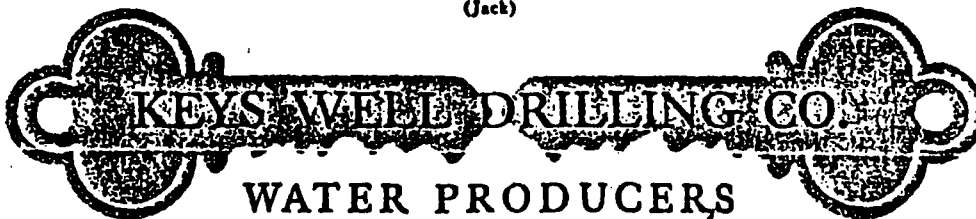
DRILLED IN NOVEMBER 1935

#2 Woodchuck

Edin A

Well
#2





Elkhurst 4998
Midway 8149

Elw. 909.33 *HB*
Power

413 No. Lexington Pkwy.
St. Paul 4, Minnesota

July 7, 1954

LOG OF WELL

VILLAGE OF EDINA, MINNESOTA

24" x 16" Well - Depth 505 feet - Water level 90 feet

96' 6" of 24" Pipe

316' of 16" liner grouted in with $31\frac{1}{2}$ cu. yds of grout

115' of 12" G.W.I pipe slotted, gravel packed

Log of Well:

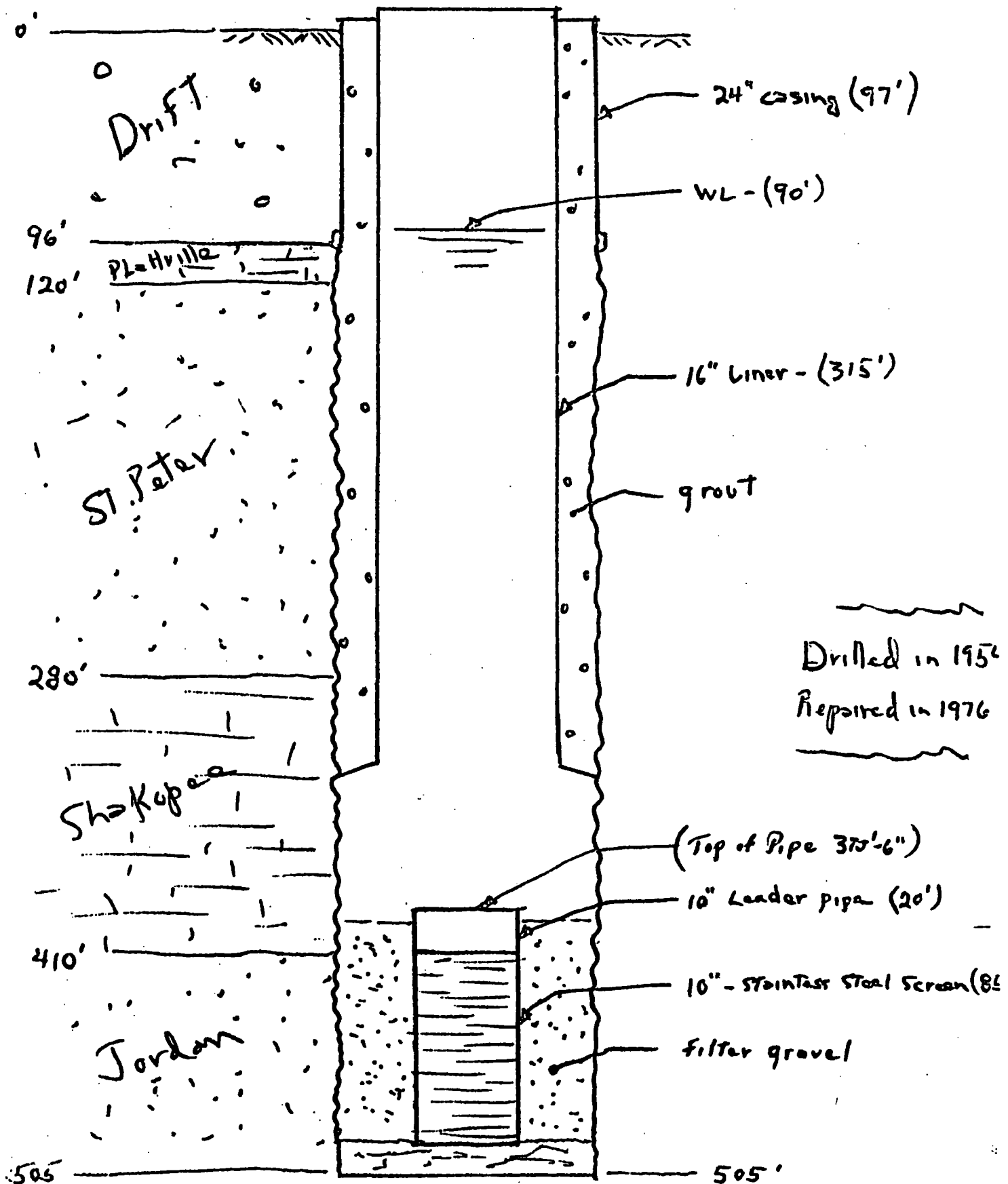
0 -	2	Pipe above ground
2 -	96	Yellow clay (sandy)
96 -	112	Limerock
112 -	120	Soapstone
120 -	280	St. Peter
280 -	410	Shakopee
410 -	495	Jordan
495 -	505	Shale

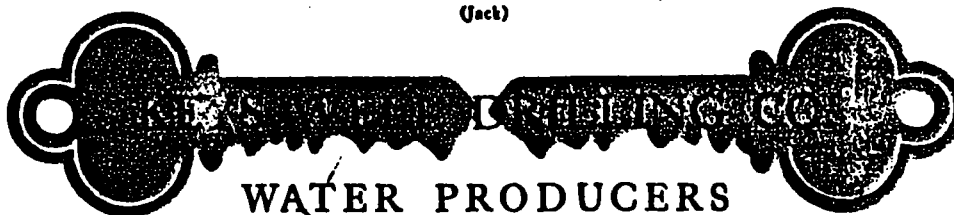
390 - FT. TOP OF SCREEN

Pumped:

<i>1956</i>	1656 G.P.M.	-	75' D.D.
	1823 G.P.M.	-	70' D.D.
	1599 G.P.M.	-	60' D.D.
	1284 G.P.M.	-	48' D.D.

WELL # 6 EDINA, Minn.





WATER PRODUCERS

Capital 7-6639
Capital 7-6630413 No. Lexington Pkwy.
St. Paul 4, Minnesota

*Sherwood Floor
EIV 853.62*

LOG OF WELLVILLAGE OF EDINA, MINNESOTAWELL #7

24" x 16" Well - Depth 547 feet - Water level 112 ft.

140' 6" of 24" Pipe

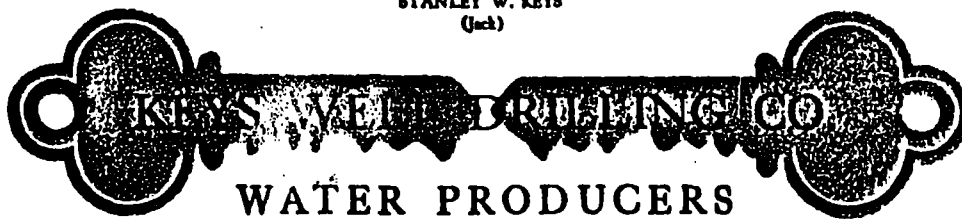
350' of 16" liner grouted in with 463 sacks cement in 1-1 mix

State 143 - 8/23/62

Tested: 1340 GPM - 25' DD
 1471 GPM - 36' DD
 1809 GPM - 47' DD

Log of Well:

0 - 18 Clay
 18 - 35 Sand and gravel
 35 - 78 Clay
 78 - 132 Sand
 132 - 159 Limerock
 159 - 162 Soapstone
 162 - 290 Sandrock
 290 - 324 Sandrock and Shale
 324 - 453 Shakopee
 453 - 545 Jordan
 545 - 547 Shale



RECEIVED
MAY 25 1964
HARRIS
ENGINE CO.

Telephones: 646-7871
646-7872

413 No. Lexington Pkwy.
St. Paul 4, Minnesota

WELL NO. 13
Belmore Park
Edina, Minnesota

24" x 16" Well 496' Deep S.W.L. 96' 7"

109' - 24" Drive Pipe
387' - 23" Open Hole
429' - 16" Liner grouted in with 44 yds. of Grout
Blasted and Bailed Well for 157½ Hours

1st TEST

1000 G.P.M. - 37' D.D.
1500 G.P.M. - 51'1" D.D.
1700 G.P.M. - 60'3" D.D.
2000 G.P.M. - 75'4" D.D.

87 Hours

BAILED WELL

101 Hours

PUMP, IN & OUT

22½ Hours

2nd TEST

2000 G.P.M. - 53'10" D.D.
1500 G.P.M. - 41'3" D.D.
1000 G.P.M. - 30' D.D.

42½ Hours

BAILED HOLE TO BOTTOM

33½ Hours

444½ Hours

Well #13

LOG:

0 - 2 Pipe above
2 - 41 Sand
41 - 60 Sandy Clay
60 - 81 Sand
81 - 106 Sandy Clay
106 - 121 Plattville
121 - 125 Soapstone
125 - 294 St. Peter
294 - 414 Shakopee
414 - 496 Jordan
496 - St. Lawrence

LAYNE-MINNESOTA CO.

FIELD REPORT OF COMPLETED WELL

Name of Job Edina Well No. 15

Date started _____

Address NON-RESPONSIVE

Date completed 11/6/67

Edina, Minnesota

No. of days _____

LOG OF WELL

ft.		Material	ft.		Material
From	To		From	To	
0	91	Sand & Clay	260	265	Hard shale, blue
91	111	Platteville rock	265	400	Rock, Shakopee
111	205	St. Peter Sandstone	400		Jordan Sandstone-shaley sand
205	210	Shale & sand mix, hard			
210	260	Shale & sand mix.			

Total - 92' 4"

Method of drilling Cable tool Rig used 28L Diam. of drill hole 30 in.

Was outer casing cemented yes Method Pump Amount cement 27 yds.

Depth of well - from ground level 405 ft. - from top of casing ft. Static 69 ft.

MATERIAL LEFT IN WELL

	Opening	Length - feet	Diameter - inches	Material
Screen		275' 10"	20" casing	3/8"
Inner casing		92' 4"	30" casing	3/8"
Outer casing		225' 3"	24" casing	3/8"

Underreamed from _____ ft. to _____ ft. Diameter _____ in. Method _____

Size-sizes-of gravel _____ Amount _____

Well shot at _____ ft. to _____ ft. depth Number _____ Size _____ lb. Material removed _____ yd.

WELL TEST

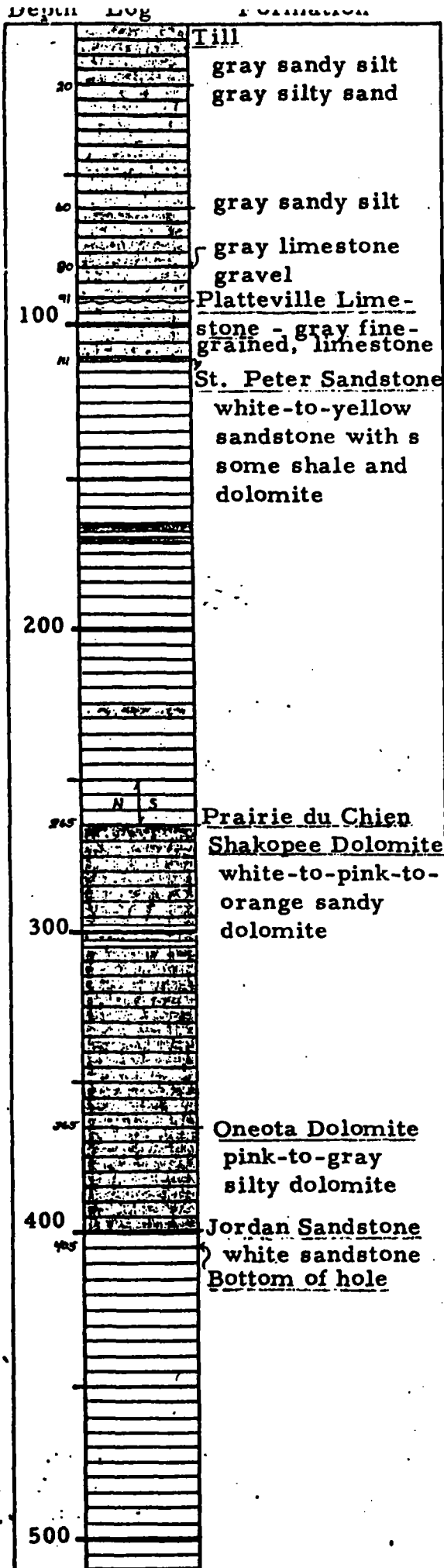
Hours Pumped		Yield gal. per min.	Water Level- ft. below surface	Drawdown feet	Remarks
From	To				
3 PM	10 PM	1200	110'	41'	Pump lot of sand & dirty water
10 PM	10 AM	2000	138'	69'	Pumping clear now & less sand
10 AM	Noon	1800	126'	57'	Least sand at this rate.
1 PM	6 PM	1500	117'	48'	No sand & clear.

Time to clear 2 days Specific Capacity 1800 g.p.m./ft. d.d. 70'

Date 11/6/67

Driller /s/ Gordon Hollen

6 bags cement for bottom plug.



Minnesota Geological Survey Minneapolis, Minnesota		
Name Edina #15		State Minnesota
Town Edina	County Hennepin	Loc.
Contractor Driller Layne-Minnesota		Sec. 29 dbb
Drilling Dates Completed 11/6/67		T. N., R. W. 117 21
Casing Record Screen - 275' 10"		
Inner casing 92' 4"		
Outer casing 225' 3"		
S.W.L. 69 ft.	G.P.M. 1800	
Remarks		Elev. 895 ± 5 ft.
		T.D.
Logged by G. S. Austin		M.G.S. No. 434

Explanation
of Colors

- Soil
- Loess, Silt on Siltstone
- Drift
- Sand & Gravel
- Shale
- Sandstone
- Limestone
- Dolomite
- Clay
- No, Samples

WELL LOG STATEMENT

File No. 0-1
Well No. 117-21-19d6d

PORT PROMPTLY TO DIRECTOR, DIVISION OF WATERS, STATE OFFICE BLDG., ST. PAUL 1, MINN.

203198
Location of Well

Hopkins Well #43
Hopkins

Locate Well on
Plat of Section

HQ

County

Describe Further

NON-RESPONSIVE

NON-RESPONSIVE

Sec.

Twp.

Range

Drilled for:

Address

Address

117-21-19A & Adde.
Elev. 915±5'

Date of Completion

1948

Date of Test

REPORT OF FINAL PUMPING TEST

Site

Upland, Valley, Hillside, Etc.

Duration of Test

Hrs.

Min.

104-B

Type of Well

Drilled
Dug, Driven, Bored, Drilled

Rate of Pumping

GPM

Drill Rig Used

Solid Tool, Jet, Rotary

Static Water Level

Ft.

450
947
109'

Diameter: Top

24"

Bottom

20"

Water Level While Pumping

Ft.

Depth of Well

775'

Drawdown

Ft.

Ground Elevation

910±

Sea Level Datum or Give Distance Above

or Below R. R., Highway, Lake, Etc.

Height of Casing Above Ground

Quality of Water

(Hard or Soft, Fresh or Salty, Etc.)

Temperature of Water

Was Laboratory Analysis Made?

For What Purpose Will Water Be Used?

Time Required for Recovery

Expected Average Yield Gal. per day

If Other Tests were Made, Give Details on Another Sheet.

Were Measurements Made of Effect on Other Nearby Wells During Test? Give Details.

No casing record

Aquifer unknown

Is Well Pumped? Pump Capacity 4250 GPM

Was Well Sealed on Completion?

Does Well Overflow Without Pumping?

Yes or No

Natural Flow GPM

What Pressure, or Head, at Ground Level?

Principal Aquifer Penetrated

Jordan S.S.

(Fill Out Both Sides of Form)

WELL LOG

915

Geologic Formations Kind, Color, Hard or Soft	Thickness of Formation	Depth in Feet		Casing Diam.	Water Conditions Found
		From	To		
Clay		1	5		CLAY
Drift { 5 ft gravel		5	45		PLTS SAND, GRVL
drift { 5 ft g.		45	80		T/835 SAND, GRVL
Plattville L.S.		80	110	OP	T/905 OPVL LMSN
Blue shale		110	116	OG	T/949' OGWD SHLE
Shale & L.S.		116	145	OSP	240 116 SNDS
St. Peter S.S.		145	225		OSTP ²⁴ SNDS
Shale Red shale		225	240		1675' 15 SHLE
Shale & Onondaga { Grey shale & L.S.		240	276	OP	SHLE, DLMT
Hard L.S.		276	396		OPDC DLMT
L.S.		396	460		DLMT
Sandy shale		460	475		CSDN SNDS

CODED

Indicate Size, Type, & Location of Any Screens, Gravel Packs, Grouting, or Other Development

I hereby certify that, to the best of my knowledge, the data presented in this statement is a true and correct representation of conditions encountered in the construction of this well.

Dated at _____ this _____ day of _____, 19__.

(Firm Name) _____

By _____

Title _____

69-2-20
16-7

MIDWEST
UNDERGROUND
INSPECTION

oute 4, Box 140 Isanti, Minnesota 55040
Robert R. Friedle 612-742-5501

WATERWELL-BOREHOLE LOG

WELL NO. 3 DATE Oct. 22, 1979

OWNER Hopkins, Minnesota

DRILLING FIRM Layne Minnesota

TYPE OF CASING Steel SIZE 16"

CASING LENGTH YEAR INSTALLED

VIDEO TAPE NO. 1

OOTAGE PHOTO NO.

CLOSED CIRCUIT TELEVISION FINDINGS

0		Pump base
27		Deflection starts
53		Deflection to 455 feet
97		Static water table
170	1	16" casing ends - Packer
174	2	Packer ends
262	3	Possible piece of casing (Can see letters printed at 10)
303		Open hole
457	4	Bottom of well.

City of Hopkins
Hopkins, Minnesota

Sketch map of well location

1. WELL DEPTH (feet)	Date of Completion
545	9/30/77

3. ☒ Cable tool 4 ☐ Power saw 7 ☐ Driven 10 ☐ Dig

☐ Hollow rod 5 ☐ Air 6 ☐ Bored 11 ☐ _____

☐ Rotary 8 ☐ Jetted 9 ☐ Power Auger

6. USE

<input type="checkbox"/> Domestic	<input checked="" type="checkbox"/> Public Supply	<input type="checkbox"/> Industry
<input type="checkbox"/> Refrigeration	<input type="checkbox"/> Air Conditioning	<input type="checkbox"/> Commercial
<input checked="" type="checkbox"/> Test Cell	<input type="checkbox"/>	

1. CALL NO.		DATE	
30	in. to 132	ft. depth	
24	in. to 354	ft. depth	
	in. to	ft. depth	

6. SERIAL _____ Or open hole
Make NONE from 354 n. to 545 n.
Type _____ Dia. _____
Plot/Gauge _____ Length _____ FEET/INCHES;
Set between _____ ft. and _____ ft.
_____ ft. and _____ ft.
_____ ft. and _____ ft.

9. STATIC WATER LEVEL
147 ft. ☒ below ☐ above Date Measured 9/29/77

150 n. after 7.516 hrs. pumping 3000 g.p.m.

☐ Fitness adapter ☐ Insect offset ☒ At least 12" above

12. Well created? ☒ Yes ☐ No Co. No. 50
1 ☒ Heat content 2 ☐ Bentonite 3 ☐ _____
Depth: from 0 ft. to 356 ft.
from _____ ft. to _____ ft.

13. Nearest source of possible contamination _____
 _____ feet _____ direction _____ type _____
 Well disinfected upon completion? Yes ☒ No ☐

14. Fuse _____

Date installed _____

☒ Not installed

Manufacturer's Name _____
Model Number _____ HP _____ Volts _____
Length of drop pipe _____ ft. capacity _____ g.p.m.
Material of drop pipe _____
Type: ☐ Fuel portable ☐ U.S. Turbine ☐ Reciprocating
☐ Jet ☐ Centrifugal ☐

16. WATER WELL CONTRACTOR'S CERTIFICATION

This well was drilled under my jurisdiction and this report is true to the best of my knowledge and belief.

Bergerson-Caswell Inc. 27058

5115 Industrial St., Maple Plain, MN.

FILED E R Kennedy DATE 10/4/77
 AUTHORIZED BY:

E. R. Henrich

1. *Journal of the American Medical Association*, 1997; 277: 1039-1043.

[illegible]

Use a second sheet, if needed.

URGENTLY FOR WITH DEED WELL OWNED COPY

KEYS WELL DRILLING COMPANY
WATER PRODUCERS
 SAINT PAUL, MINNESOTA

SAINT PAUL, MINNESOTA

WATER PRODUCERS

SAINT PAUL, MINNESOTA

Owner VILLAGE OF MINNETONKA

Date ~~SECRET~~ Partial May 2, '67

Location **NON-RESPONSIVE**

Driller Sittig

Well No. 6 Size 24 x 20 x 16 Total Depth 485' Type Jordan

DRILLERS LOG

0	to 101	Drift
101	to 117	Limerock
117	to 133	Shale
133	to 240	Sandrock (soft)
240	to 261	Shale
261	to 310	Sandrock (soft)
310	to 322	Shakapan (broken)
322	to 392	Shakapan (sandy)
392	to 435	Jordan (soft)
435	to 442	Jordan (shale)
442	to 465	Jordan
465	to 481	Shale
	to	
	to	

PERMANENT PUMP DATA

Mfg. _____ Type _____ Serial No. _____
Capacity _____ GPM _____ TDH _____
Motor Make _____ Type _____
_____ H. P. _____ Volts _____ Ph. _____ RPM _____
_____ ft. _____ in Col. pipe _____ in. Shaft _____
_____ ft. _____ in Bowls _____ Stages _____ Type _____
_____ ft. _____ in suction pipe & _____
_____ ft. Total Length of Pump _____
_____ ft. _____ in. drop pipe & _____ No. Cable _____
_____ ft. _____ in. air line _____
_____ in. Pitless _____ ft. bury _____ in outlet _____

WELL MATERIALS

103 ' of 24 " diameter of Outer Casing
52 ' of 23 " diameter of Open Hole
322 ' of 20 " diameter of Inner Casing
166 ' of 19 " diameter of Open Hole
52 ' of 16" liner from (312 to 354)
354 ' to 0 Mix grout 570 (300) (Sacks)
" diameter _____ Screen

RECORD OF TEST PUMPING

Static Water Level _____ ft. from _____
 _____ GPM _____ D.D. _____ Hours
 363 GPM 48' 8" D.D. 2 Hours
 470 GPM 54' 9" D.D. 5 1/2 Hours
 633 GPM 69' 8" D.D. 2 Hours
 _____ GPM _____ D.D. 1 1/2 Hours

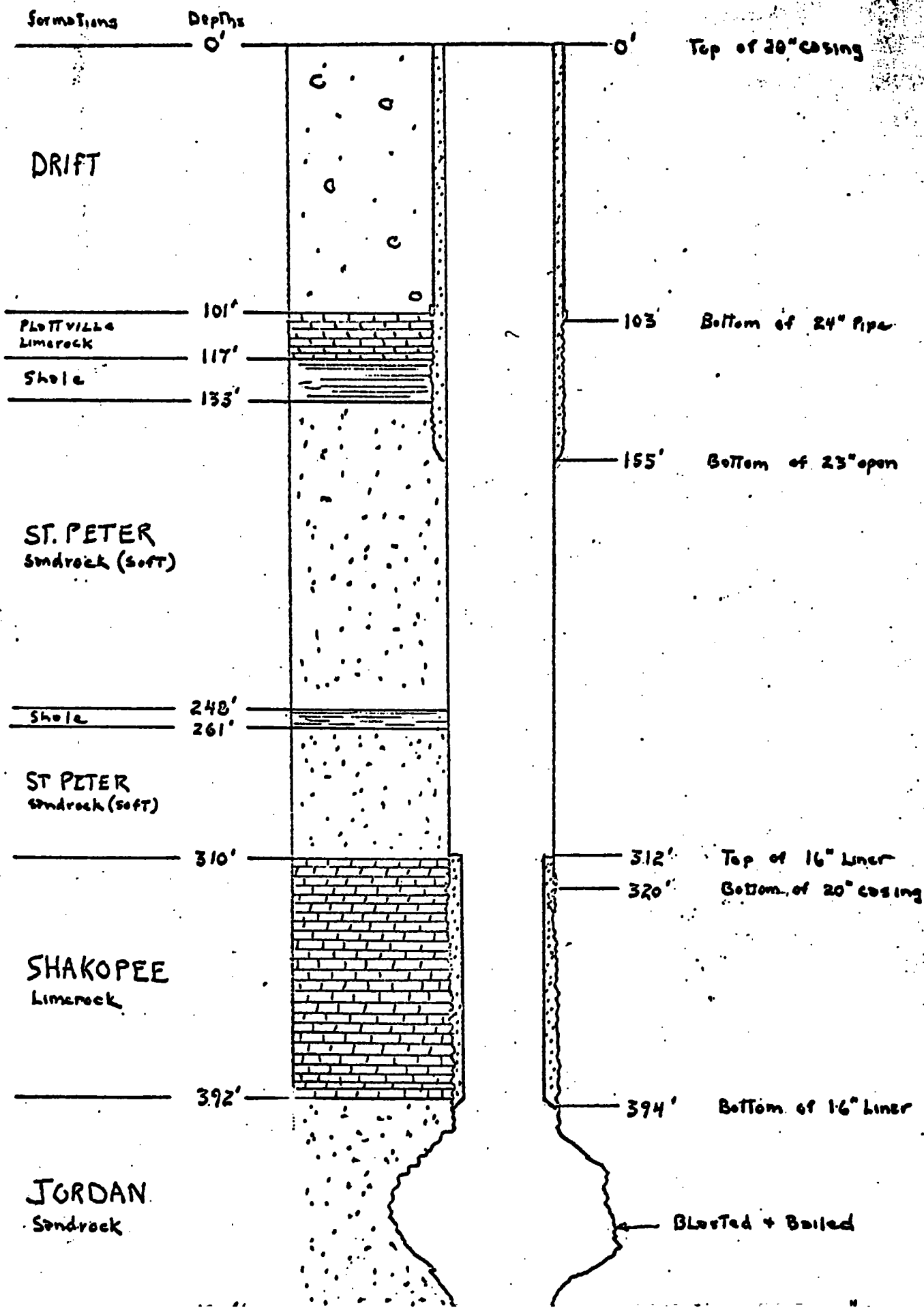
Remarks: After its 1st test - 16" liner
was installed from 312' to 334' which
cut off the Shakamon formation
and then the well is being blasted
and bailed in the lower part of the
Jordan formation

[illegible]

WELL LOG - INNENONKA VILLAGE WELL #6

ENGINEER - Schoell & Madson

CONTRACTOR - KEYS Well Drilling Co.



KEYS WELL DRILLING COMPANY

WATER PRODUCERS

SAINT PAUL, MINNESOTA

Owner VILLAGE OF MINNETONKADate Completed Partial 5/2/67Location NON-RESPONSIVEDriller Floyd O'BrienWell No. 7 Size 24 x 20 Total Depth 334(?) Type ?

DRILLERS LOG

0 ' to 104 ' Drift
104 ' to 112 ' Shale
112 ' to 121 ' Limerock
121 ' to 136 ' Shale
136 ' to 249 ' Sandrock (soft)
249 ' to 261 ' Shale
261 ' to 304 ' Sandrock
304 ' to 315 ' Shakopco (broken)
315 ' to 339 ' Shakopco (sandy)
 ' to '
 ' to '
 ' to '
 ' to '
 ' to '
 ' to '

PERMANENT PUMP DATA

Mfg. Type Serial No.
 Capacity GPM TDH
 Motor Make Type
 H. P. Volts Ph. RPM
 ft. in Col. pipe in. Shaft
 ft. in Bowls Stages Type
 ft. in suction pipe &
 ft. Total Length of Pump
 ft. in. drop pipe & No. Cable
 ft. in. air line
 in. F. less ft. bury in outlet

WELL MATERIALS

114 ' of 24 " diameter of Outer Casing
38 ' of 23 " diameter of Open Hole
315 ' of 20 " diameter of Inner Casing
24 ' of 20 " diameter of Open Hole
 ' to Mix grout (yds.) (Sacks)
 ' " diameter Screen

RECORD OF TEST PUMPING

Static Water Level ft. from
 GPM D.D. Hours
 GPM D.D. Hours
 GPM D.D. Hours
 GPM D.D. Hours
 GPM D.D. Hours

Remarks: Drilling was stopped at 339'
because of sandrock running into
hole. Hole was bailed to 322' and filled
with 70 sacks of concrete and then
drilled out and when we reached 339'
again it again filled back up to 325'.

WELL LOG , INNENETONKA VILLAGE WELL #7

ENGINEER - Schoell & Madson

Contractor - KEYS Well Drilling Co.

Formations

Depths

Depths

0' Top of 20" casing

DRIFT

PLATTVILLE
Limerock
Shale
104'
112'
136'

ST PETER
Sandrock (soft)

Shale
249'
261'

ST. PETER
Sandrock (soft)

304'

SHAKOPEE
Limerock (Broken 315')

392'

JORDAN
Sandrock

85'8" Static water level

114' Bottom of 24" casing

152' Bottom of 25" open hole

285' Top of 12" Liner

299' Top of 16" Liner

315' Bottom of 20" pipe

341' Bottom of 16" Liner

397' Bottom of 12" Liner

Blasted & Boiled

REPORT

Tack No. _____

Well No. _____

223938

Town. Minneapolis 28/24-5 kab

Date Started. _____ Machine No. _____ State. Minnesota

Date Completed. _____ Owner. American Hardware Mutual Ins.

Location. 3033 Excelsior Blvd. Total Depth of Well. 520'

DIAMETER OF HOLE	16"	12"	10"	PA
Top of Pipe above Surface	16'	92'	16'	75-616
Bottom of Pipe below Surface	102'9"	204'	284'	
No. of Ft. of Pipe in the Hole	85'9"	112'	268'	
No. of Ft. of Hole Drilled				

TEST	1	2	3	4	FORMATION	Thickness	Depth
Depth of the Hole.....	520				Sand OF U - SAND	18	0-12
Depth to Water at Rest	60				Sand & Gravel OF U - SAND, GRV	15	8-33
Depth to Water Pumping	66				Sand (Wet) OF U - SAND	14	23-47
Depth of Pump Pipe	700				Blue Clay OF U - CLAY	6	41-53
Gallons per minute	Yes				Water Sand OF U - SAND	43	53-96
Will well supply more?	Yes				Sand & Gravel - Broken	19	115
Was Strainer in Hole?	Yes				OF U - SAND, GRV, Lime	13	128
Was water clear?	Yes				Sand OF U - SAND	47	175
Was well pumping sand?					Sand & Gravel OF U - SAND, GRV	20	195
Hours Pumping					Gravel - S off Sandrock	15	210
					Yellow Shale & Sandrock	60	270
					Sandrock & Shale OF U - SAND	20	391
					Limestone OF U - PLAT	129	520
					Sandrock OF U - SAND		

STRAINER

Make	
Type of Metal	
Diameter O. D.	
Diameter I. D.	
Total Length	
Number	
Top of Screen below Surface	
No. of Ft. Exposed	
Bottom of Screen below Surface	
Was Str. welded	
Did Sand come thru Str.	
Was Str. coarse enough	
Style of Fittings	

All measurements taken from Drilling

Drilling Platform 18' above boiler room floor.

885
195
690 - 40TP

270
175 - T/OPDC

391
194 - 40DN

LOCATED BY

- ☐ Address Verification
- ☐ Name on Mailbox
- ☐ Lot-Block
- ☐ Plat Book
- ☐ Info. From Owner
- ☐ Info. From Neighbor
- ☒ Other Superintendent
- ☐ Can't Locate State Why

9630359

APPENDIX C
CONTINGENCY PLAN

QUALITY ASSURANCE BRANCH

APR 05 1988

ENVIRONMENT SERVICES DIVISION

Contingent Actions for Contaminated Water

It is possible that groundwater contaminated with coal tar materials will be encountered during the sample retrieval operations. Groundwater generated during sample retrieval operations will be classified as contaminated if the water exhibits a discernible oil sheen or oil phase. Contaminated water will be pumped to the sanitary sewer if it contains less than ten percent organic material. Estimates of flow rate, disposal volume and water quality will be established and the Metropolitan Waste Control Commission (MWCC) will be informed before the discharge to the sanitary sewer if the estimated flow exceeds 150 gallons per workday from any individual site. Contaminated liquids containing more than ten percent organic material or failing to receive MWCC approval for discharge will be disposed of in accordance with all applicable local, state and federal rules and regulations and Part T of the Consent Decree. Uncontaminated water will be disposed of in the storm sewer or by other means acceptable to the City of St. Louis Park.

The City will be responsible for keeping the Environmental Protection Agency, Minnesota Pollution Control Agency and Reilly Tar & Chemical Corporation informed of all significant actions involving the generation of contaminated groundwater. All actions, decisions and communications by the City, Environmental Protection Agency, Minnesota Pollution Control Agency, and Reilly in dealing with contaminated soils will be in accordance with and subject to the provisions of Parts I, J, and O of the Consent Decree in the Reilly settlement.

QUALITY ASSURANCE PROJECT PLAN

Page: 1 of 68
Date: Jan. 1988
Number: RAP 3.2.
Revision: 2

QUALITY ASSURANCE PROJECT PLAN
FOR SAMPLING AND ANALYSIS - GROUNDWATER
AND GAC PLANT MONITORING

Prepared by

The City of St. Louis Park
St. Louis Park, MN 55416

Approved by: Robert C. Hanisch Date: February 16, 1988
Robert Hanisch, Quality Assurance
Director, Rocky Mountain Analytical Laboratory

Approved by: James N. Grube Date: March 30, 1988
James N. Grube, Project Manager
City of St. Louis Park, MN

Approved by: Andrea Jirka Date: 5/11/88
Andrea Jirka, Quality Assurance
Officer; U.S.EPA, Region V

Approved by: _____ Date: _____
Erin Moran, Remedial Program Manager,
U.S. EPA Region V

QUALITY ASSURANCE BRANCH

APR 05 1988

ENVIRONMENT SERVICES DIVISION

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APPENDIX A - STANDARD OPERATING PROCEDURES

APPENDIX B - METHOD DETECTION LIMIT STUDIES/STANDARD OPERATING PROCEDURE

3. PROJECT DESCRIPTION

3.1 Background

Groundwater in the city of St. Louis Park, Minnesota has been contaminated by activities at a coal-tar distillation and wood preserving plant operated from 1917 to 1972. Numerous previous studies have identified polynuclear aromatic hydrocarbons (PAH) present in various aquifers beneath St. Louis Park and adjacent communities.

The United States Environmental Protection Agency (EPA), the Minnesota Pollution Control Authority (MPCA), the Minnesota Department of Health (MDH), the City of St. Louis park (SLP), and Reilly Tar & Chemical Corporation (Reilly) have agreed to acceptable water quality criteria for PAH. These criteria, as incorporated into the Consent Decree - Remedial Action Plan (RAP), include the following concentration levels:

	<u>Advisory Level</u>	<u>Drinking Water Criteria</u>
o Sum of benzo(a) pyrene and dibenz(a,h) anthracene	3.0 ng/l*	5.6 ng/l
o Carcinogenic PAH	15 ng/l	28 ng/l
o Other PAH	175 ng/l	280 ng/l

*or the lowest concentration that can be quantified,
whichever is greater

In conjunction with the implementation of remedial measures to limit the spread of contaminants, a granular activated carbon (GAC) treatment system has been installed to treat water from St. Louis Park (SLP) wells 10 and 15. Further provisions of the Remedial Action Plan (RAP) call for long-term monitoring of the influent and effluent of the GAC treatment plant and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH and/or phenolics in the ground water. The analytical data will be used to evaluate contamination by comparing the levels of PAH and/or phenolics found in the various samples with historical water quality data and with water quality criteria established in the Consent Decree-RAP. The specific objectives of the sampling and analysis program, and therefore, the intended end use of the data vary slightly for the different aquifers (Mt. Simon-Hinckley, Iron-ton-Galesville, Prairie du Chien-Jordan, St. Peter, and Drift- Platteville) being monitored in accordance with the Consent Decree-RAP.

3.2 Objectives and Intended Data Usage

The GAC plant monitoring is being done to assess and continuously evaluate the performance of the treatment system. Analytical results for influent and effluent samples will be compared to the drinking water criteria for PAH as established in the Consent Decree-RAP. Based on these comparisons, decisions will be made on: 1) possible modifications to the treatment system (e.g., adding another carbon column), 2) system operations (e.g., when the carbon should be replaced), and 3) cessation of the treatment system, if desired, when sufficiently low concentrations of PAH in influent samples are demonstrated.

The objective of sampling the four existing Mt. Simon-Hinckley Aquifer municipal drinking water wells, and sampling any new Mt. Simon-Hinckley Aquifer municipal drinking water wells installed within one mile of well W23, and analyzing for PAH is to assure the continued protection of these wells from PAH resulting from activities of Reilly at the site. The analytical data will be used to make comparisons between the levels of PAH found in the Mt. Simon-Hinckley Aquifer, and the drinking water criteria established in the Consent Decree-RAP.

The objective of sampling and analyzing the Ironton-Galesville Aquifer source control well (W105) is to assess the levels of PAH in the discharge from W105 when it is pumping a monthly average of 25 gallons per minute. The data will be used to compare the concentration of total PAH in the samples to a cessation criterion of 10 micrograms per liter of total PAH established in the Consent Decree-RAP. Also, if any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23, then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the well from PAH resulting from the activities of Reilly at the site. The analytical data would be used to compare the levels of PAH found in potential Ironton-Galesville Aquifer drinking water wells to the drinking water criteria established in the Consent Decree-RAP.

The objectives of monitoring the many Prairie du Chien-Jordan Aquifer wells, including municipal drinking water wells, private or industrial wells, and monitoring wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control system, and 2) assure the continued protection of drinking-water wells from PAH resulting from the activities of Reilly at the site. The analytical data will be used to compare the levels of PAH in the Prairie du Chien-Jordan Aquifer to historical PAH data and to various criteria established in the Consent Decree-RAP (e.g., drinking water criteria for drinking water wells, and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23). Analytical data for samples of the discharge from gradient control well SLP4 will be compared to discharge limitations in an NPDES permit which will be applied for at the conclusion of a Feasibility Study to determine the appropriate disposition of SLP4 discharge. Water level data will be used to evaluate ground-water flow patterns in the Prairie du Chien-Jordan Aquifer.

The objective of monitoring St. Peter Aquifer wells is to determine the nature and extent of PAH in the St. Peter Aquifer resulting from the activities of Reilly at the site. The analytical data will be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data and to the drinking water criteria established in the Consent Decree-RAP. Water level data will be used to evaluate ground-water flow patterns in the St. Peter Aquifer.

The objectives of monitoring the Drift-Platteville Aquifer wells are to: (1) monitor the distribution of PAH and phenolics in the aquifer, thus evaluating the source and gradient control systems, and (2) to further define the nature and extent of PAH and phenolics in the Northern Area of the Drift-Platteville Aquifer resulting from the activities of Reilly at the site. The analytical data will be used to compare levels of PAH and phenolics in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the Consent Decree-RAP for PAH and phenolics. Water level data will be used to evaluate ground-water flow patterns in the Drift-Platteville Aquifer.

The Site Management Plan outlines the scope of work to be performed in order to monitor the ground water in the St. Louis Park, MN area in accordance with the Consent Decree-RAP related to the Reilly Tar & Chemical Corp. N.P.L. site. Included in this plan are: (1) the identity of wells to be monitored, (2) the schedule for ground-water monitoring, and (3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting.

The time period covered by the Initial Sampling Plan is from the date of its acceptance and approval by the agencies, to December 31, 1988. The first subsequent Sampling Plan (RAP Section 3.3) will be submitted by October 31, 1988, covering the 1989 calendar year.

This Plan incorporates the requirements of RAP Sections 3.2, 3.3, 4.3, 5.1, 6.1.4, 7.3, 8.1.3, 9.1.3, 9.2.3, 9.3.3, and 9.6. Some of the sampling required under RAP Section 4.3 (Monitoring the GAC System) has already taken place prior to the Effective Date. Therefore, only the monitoring that will take place from the approval date of this Initial Sampling Plan through December 31, 1988 is included in this Plan. Section 9.3.3 of the RAP enables the EPA Regional Administrator, the Director of the MPCA or the Commissioner of Public Health to request expanded analyses (including volatiles, acids, base/neutrals, metals, ammonia, chloride, sodium and sulfate). Should these analyses be required, an addendum will be written to the QAPP which will encompass all methodological references.

4. PROJECT ORGANIZATION AND RESPONSIBILITIES

This project is being conducted in accordance with the Consent Decree Remedial Action Plan for the Reilly Tar & Chemical Corporation N.P.L site in St. Louis Park, Minnesota. The parties to the Consent Decree include Reilly, the City of St. Louis Park, U.S. EPA, MPCA, and MDH. The project organization shown in Figure 4-1 indicates the involvement of the parties to the Consent Decree, as appropriate. The City shall be assisted by two consultants in the retrieval and laboratory analysis of water samples.

Environmental Research Technology, Inc. (ERT) will be responsible for the coordination of all field sample retrieval and Rocky Mountain Analytical Laboratory (RMAL) with analytical facilities in Arvada, Colorado, shall be responsible for the coordination and completion of all laboratory analyses. Responsibilities of the key positions in the organization of RMAL are described below:

- o Laboratory Project Manager: The Laboratory Project Manager's responsibilities include coordination of activities, project communication, and general overview of the program progress.
- o Laboratory Director: The Laboratory Director is responsible for scheduling personnel and resources to meet project commitments.
- o Operations Manager: The Operations Manager is responsible for oversight of preparation and analysis of PAH samples to ensure that project objectives, requirements and QA/QC criteria are met.
- o Laboratory Supervisor: Laboratory Supervisor shall be responsible for daily supervision of technicians and analysts for PAH and total phenolics analyses.
- o Preparation Supervisor: The Preparation Supervisor is responsible for oversight of sample extraction and preparation for analysis.
- o Analyst: The Analyst is responsible for the analysis of water samples for the requested parameters utilizing the methods prescribed by this Plan.
- o Technician: The Technician is responsible for sample extraction. This requires practical experience and knowledge in the techniques of liquid - liquid solvent extraction, Kuderna - Danish evaporation, and the quantitative preparation of sample extracts for analysis.

- o **Quality Assurance Director:** The Quality Assurance Director is responsible for overall quality control oversight. The Quality Assurance Director supervises an independent QA/QC department and reports directly to the Division Director and Corporate Vice President for Quality Assurance.
- o **Sampling Team:** The Sampling Team shall consist of employees of the City of St. Louis Park and ERT. The team shall be responsible for sample collection; conducting field measurements (i.e. water level); and maintaining proper decontamination procedures stated in the Quality Assurance Project Plan.
- o **Data Assessment:** The evaluation of data, as it is compiled and organized in accordance with the requirements of the Quality Assurance Project Plan, is the responsibility of the Operations Manager. Additional review, evaluation, and assessment of the data is performed by the Laboratory Manager, thereby providing additional assurance that the requirements of the Quality Assurance Project Plan are met.
- o **The EPA Contract and Program Management Section (CPMS), Region V,** shall be responsible for the review of up to 10 percent of the reports and data packages generated in accordance with Section 10.3. of this Quality Assurance Project Plan.
- o **Performance and System Audits:** The Contract Project Management Section (CPMS) of Region V, Central Regional Laboratory (CRL) is responsible for both Performance and System audits of the laboratory selected for this project.

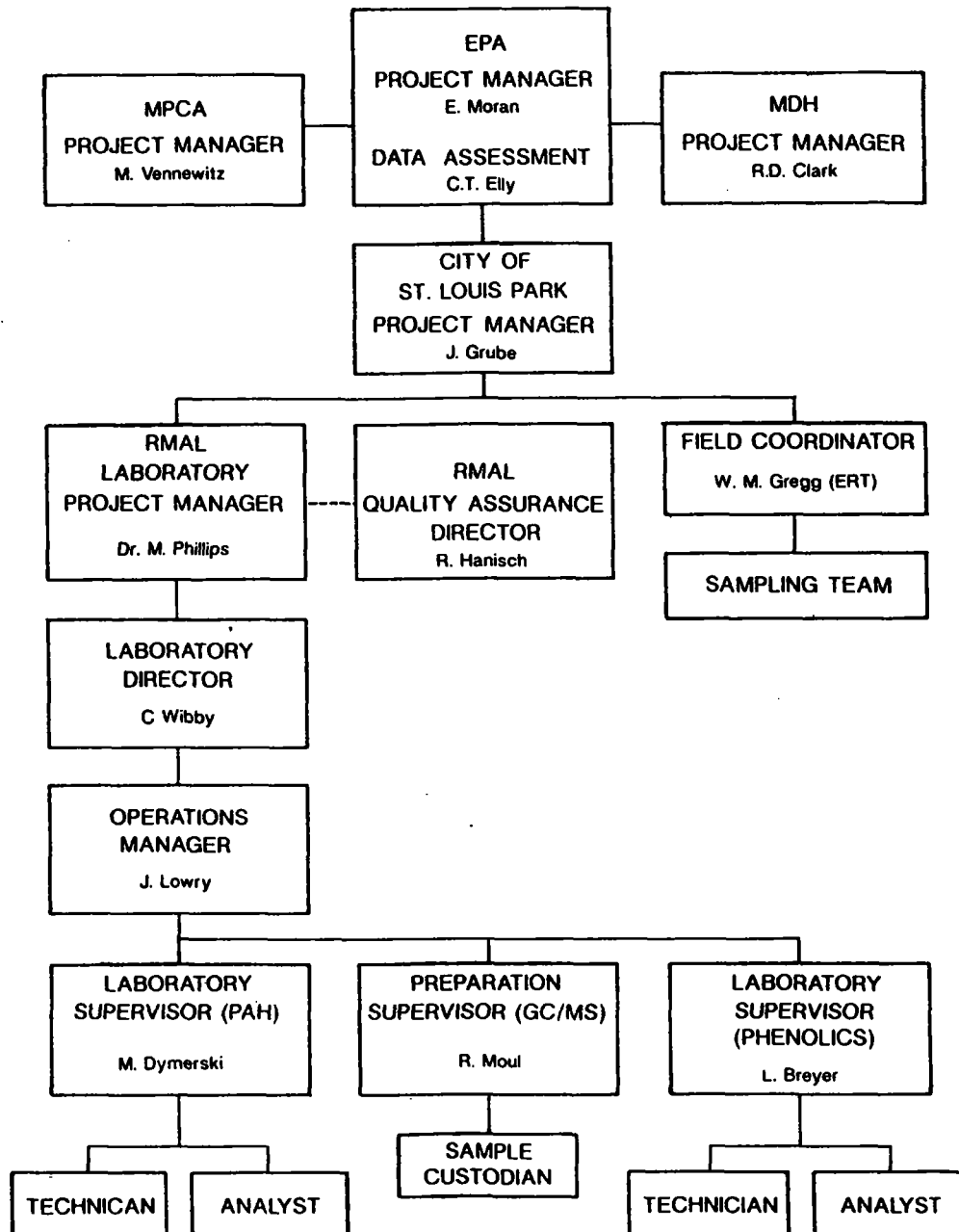


Figure 4-1 Project Organizational Chart

5. QUALITY ASSURANCE OBJECTIVES

The principal objectives of this Plan pertain to the collection of data that are sufficient to monitor the effectiveness of the GAC treatment system and to detect changes in groundwater quality. Therefore, the quality of the data gathered in this project can be defined in terms of the following elements:

- o Completeness - a sufficient number of successful (valid) measurements to characterize the concentrations of PAH in the influent and effluent of the treatment system and in the aquifers of interest over a period of time.
- o Representativeness - the extent to which reported analytical results truly depict the PAH concentrations in the sampled environment. Representativeness is optimized through proper selection of sampling sites, times and procedures, through proper sample preservation, and through prompt extraction and analysis.
- o Accuracy and Precision - Accurate and precise data will be achieved through the use of sampling and analytical procedures that minimize biases, through the use of standard procedures, through the meticulous calibration of analytical equipment and by implementing corrective action whenever measured accuracy and precision exceed pre-established limits. Accuracy and precision will be measured by the analysis of method spikes and duplicate samples.
- o Sensitivity - determination of instrument sensitivity is accomplished by calibration using multiple concentrations of the analytes of interest. Once instrument sensitivity is demonstrated, analysis of replicate spiked samples of deionized reagent water at a concentration of 1-5 times the instrument sensitivity, is used to determine method sensitivity (i.e. method detection limit)
- o Comparability - the extent to which comparisons among separate measurements will yield valid conclusions. Comparability among measurements in the SLP monitoring program will be achieved through the use of rigorous standard sampling and analytical procedures.
- o Traceability - the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis.

The fundamental mechanisms that will be employed to achieve these quality goals can be categorized as prevention, assessment and correction, as follows:

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- 1) Prevention of defects in the quality through planning and design, documented instructions and procedures, and careful selection and training of skilled, qualified personnel;
- 2) Quality assessment through a program of regular audits and inspections to supplement continual informal review;
- 3) Permanent correction of conditions adverse to quality through a closed-loop corrective action system.

The St. Louis Park sampling program Quality Assurance Project Plan has been prepared in direct response to these goals. This Plan describes the quality assurance program to be implemented and the quality control procedures to be followed by RMAL during the course of laboratory analyses in support of the various site investigation studies for the St. Louis Park (SLP) site. The QA objectives will include field blanks, method blanks, field duplicates, surrogate spikes, and matrix spikes. Precision, accuracy and completeness criteria are established for each parameter of interest. The specific criteria for each analysis and parameter are set forth in detail in the following sections:

<u>Objective</u>	<u>Frequency</u>	<u>Sections Discussing Criteria</u>
Field Duplicates	10%	6.8, 11.1.5
Field Blanks	10%	6.5.2
Method Blanks	5%	11.1.2
Surrogate Spikes	100% of GC/MS analyses	11.1.3, 15
Matrix Spikes	5%	11.1.4, 15

6. SAMPLING PROCEDURES

Samples will be collected by ERT and SLP personnel. The overall sampling program is summarized in Tables 6-1 and 6-2, and Figures 6-1 through 6-5. This section discusses general QAPP provisions relevant to sample collection, containerization, packaging and shipping activities.

6.1 Training

All ERT and SLP personnel working on the project will be properly trained, qualified individuals. Prior to commencement of work, personnel will be given instruction specific to this project, covering the following areas:

- o Organization and lines of communication and authority
- o Overview of the Site Management Plan and QA Project Plan,
- o Documentation requirements,
- o Decontamination requirements,
- o Health and Safety considerations.

Training of field personnel will be provided by the Field Coordinator or his/her qualified designee.

The analysts performing chemical analyses of samples will be trained in and will have exhibited proficiency in the analytical methods to be employed.

6.2 Document Control

Document Control for the Initial Sampling Plan serves a two-fold purpose. It is a formal system of activities that ensures that:

- 1) All participants in the project are promptly informed of revisions of the Quality Assurance Project Plan; and
- 2) All documents generated during the course of the program are accounted for during, and at the end of the project.

This QA Project Plan and all Standard Operating Procedure documents have the following information on each page:

- o Document Number
- o Page Number
- o Total number of pages in document
- o Revision number
- o Revision date

TABLE 6-1
INITIAL SAMPLING PLAN GAC PLANT
MONITORING SCHEDULE^(a)

<u>RAP Section</u>	<u>Sampling Points</u>	<u>Start of Monitoring</u>	<u>Sampling Frequency</u>	<u>Analyses^(b)</u>
4.3.1(C)	Treated water(TRTD)	Date of plan approval	Monthly	PAH(ppt) ^(c)
4.3.3(C)	Feed water(FEED)	Date of plan approval	Quarterly	PAH(ppt)
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH(ppt)
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds in EPA Test Method 625.

- (a) This schedule does not include certain contingencies (eg. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1988. Sections 4 and 12 of the RAP outline the additional sampling that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. The location of the GAC plant is shown in Figure 6-1.
- (b) List of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds in EPA Test Method 625 are provided in the QAPP. Field blanks will be collected and analyzed at a frequency of one per day or one per 10 samples, whichever is more frequent. Treated water will be duplicated at a rate of 100%. For the feed water duplicate samples will be collected and analyzed at a frequency of one per 10 samples.
- (c) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

TABLE 6-2
INITIAL SAMPLING PLAN GROUNDWATER
MONITORING SCHEDULE(a)

<u>RAP Section</u>	<u>Source of Water</u>	<u>Sampling Points</u>	<u>Start of Monitoring</u>	<u>Sampling Frequency</u>	<u>Analyses</u>	<u>Duplicate Samples</u>
5.1	Mt. Simon- Hinckley Aquifer	SLP11, SLP12, SLP13, SLP17	Within six months of Effective date ⁽⁹⁾	Annually	PAH(PPT) ^(c)	SLP11, SLP12, SLP 13, SLP 17
5.3.2		New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)	
6.1.4	Ironton- Galesville Aquifer	W105 W38 ^(e)	Start of pumping	Quarterly	PAH(ppb) ^(d)	
6.2.1.		New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)	
7.3(A)	Prairie du Chien- Jordan Aquifer	SLP4	Start of pumping	Quarterly	PAH(ppt) ^(h) total phenols	SLP4
7.3(B)		W23	Start of pumping	Quarterly	PAH(ppb)	W23
7.3(C)		SLP6, SLP7 or SLP9, W48	Date of plan approval	Quarterly	PAH(ppt)	SLP6, SLP7 or SLP9
7.3(D) ^(m)		AHM or MGC ⁽ⁱ⁾ , E2, E13, H3, SLP10 or SLP15, SLP14, SLP16, W402 ^(j) W403, W119	Date of plan approval	Semi-annually	PAH(ppt)	
7.3(E) ^(m)		SLP5, H6, E3, E15, MTK6, W29, W40, ^(j) W70, W401 ^(j)	Date of plan approval	Annually	PAH(ppt)	SLP5, H6, E3, E15 MTK6
7.3(F)		W112, W32, SLP8, SLP10, E4, E7	Date of plan approval	Quarterly	No chemical analyses ^(f)	

TABLE 6-2 (Continued)

<u>RAP Section</u>	<u>Source of Water</u>	<u>Sampling Points</u>	<u>Start of Monitoring</u>	<u>Sampling Frequency</u>	<u>Analyses</u>	<u>Duplicate Samples</u>
8.1.3	St. Peter Aquifer	SLP3, W14, W24, W33, W122, W129, W133, P116, plus 5 new wells (A,B,C,D,E)	Within 30 days of installing new wells	Once	PAH(ppt)	SLP3, WC
		SLP3 plus six of the wells listed above ^(h)	Within 6 months of above	Once	PAH(ppt)	SLP3
9.1.3 and 9.2.3	Drift-Platteville Aquifer	Source and gradient control wells (3 wells)	Start of pumping	Quarterly	PAH(ppb) and total phenols	
9.3.3		W131, W136, plus 6 new wells	Within 30 days of well installations	Once	PAH (ppb) and total phenols	New Drift Well W. 35th St.
9.3.3		W131, W136, plus 6 new wells	Within 6 months of above	Once	PAH (ppb) and total phenols	New Drift Well W. 35th St.
9.6		Drift: W2, W6, W10, W11, W12, W116, W117, W128, W135, W136, P109, P112' Platteville: W1, W18, W19, W20, W27, W101, W120, W121, W124, W130, W131, W143, plus 6 new wells	Concurrent ^(k) with 9.3.3 sampling	Concurrent ^(k) with 9.3.3 sampling	PAH(ppb) and total phenols	New Drift Well W. 35th St. W10

- (a) This schedule does not include certain contingencies (eg. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1988. Section 12 of the RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. Field blanks will be collected at a frequency of one per day or one per 10 samples whichever is more frequent. Duplicate samples will be collected as noted, or at least one for every 10 samples.

TABLE 6-2 (Continued)

- (b) Lists of parameters and descriptions of the methods for analysis of PAH, phenolics, and expanded analyses are provided in the QAPP. Water levels will be measured each time samples are collected for analysis, except for those wells which prove to be inaccessible for such measurements.
- (c) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.
- (d) ppb = parts per billion. This signifies analysis by EPA Method 625. If analytical results for individual wells are below 20 micrograms per liter (20 ppb) using this method, then the part per trillion method will be used on subsequent monitoring rounds.
- (e) Water levels in W38 will be measured each time W105 is sampled.
- (f) Water levels only (no monitoring) will be measured at these wells, except for those wells which prove to be inaccessible for such measurements.
- (g) Or within 30 days of the approval date of the Plan, whichever is later.
- (h) SLP4 analytical program will be determined by the results of the Feasibility Study.
- (i) AHM = American Hardware Mutual, MGC = Minikahda Golf Course.
- (j) Wells W401, W402, and W403 may or may not be available for sampling at the same time as the other wells on these lists. They will be sampled in conjunction with the monitoring performed in accordance with the schedule shown, once they are available for sampling.
- (k) If any of the wells listed here become damaged, destroyed, or otherwise unsuitable for sampling, alternate wells will be selected by the Project Leaders for monitoring.
- (l) Sampling points are located on the maps shown in Figures 6-1 through 6-5. Letter prefixes to well codes are defined as follows (W23, W48, W105, source control and gradient control wells will be sampled at discharge head):
 - W - 4-inch monitoring well (at top of casing)
 - P - monitoring piezometer (at top of casing)
 - SLP - St. Louis Park supply well (at discharge head)
 - E - Edina supply well (at discharge head)
 - H - Hopkins supply well (at discharge head)
 - MTK - Minnetonka supply well (at discharge head)
- (m) Water level measurements will be made quarterly at these wells, except for those wells which prove to be inaccessible for such measurements.
- (n) The six St. Peter Aquifer monitoring wells that will be monitored according to RAP Section 8.1.3 will be elected by the Project Leaders based on the results of the first monitoring round.

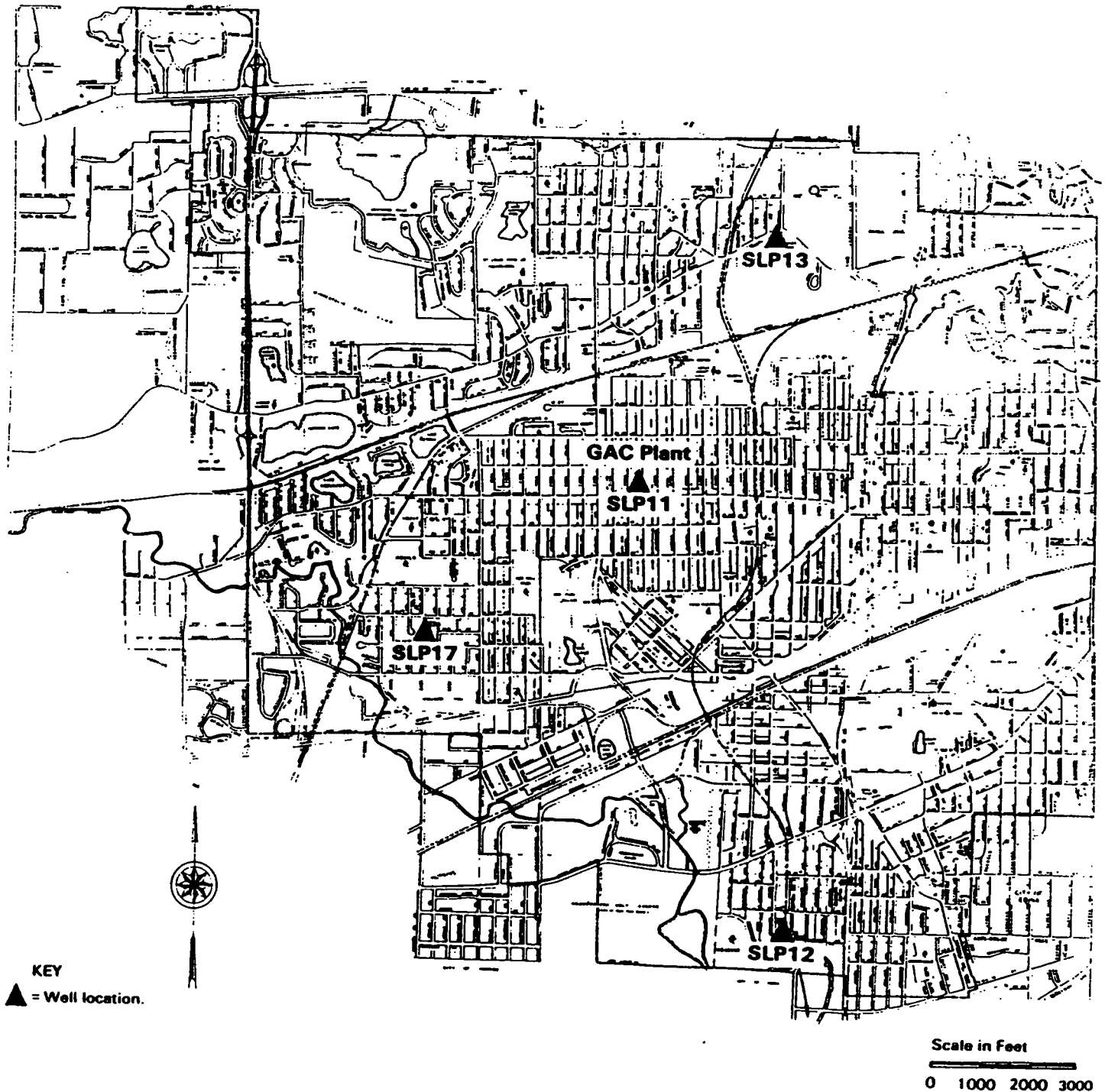


Figure 6-1 Location of Mt. Simon-Hinkley Monitoring Wells and St. Louis Park GAC Water-Treatment Plant

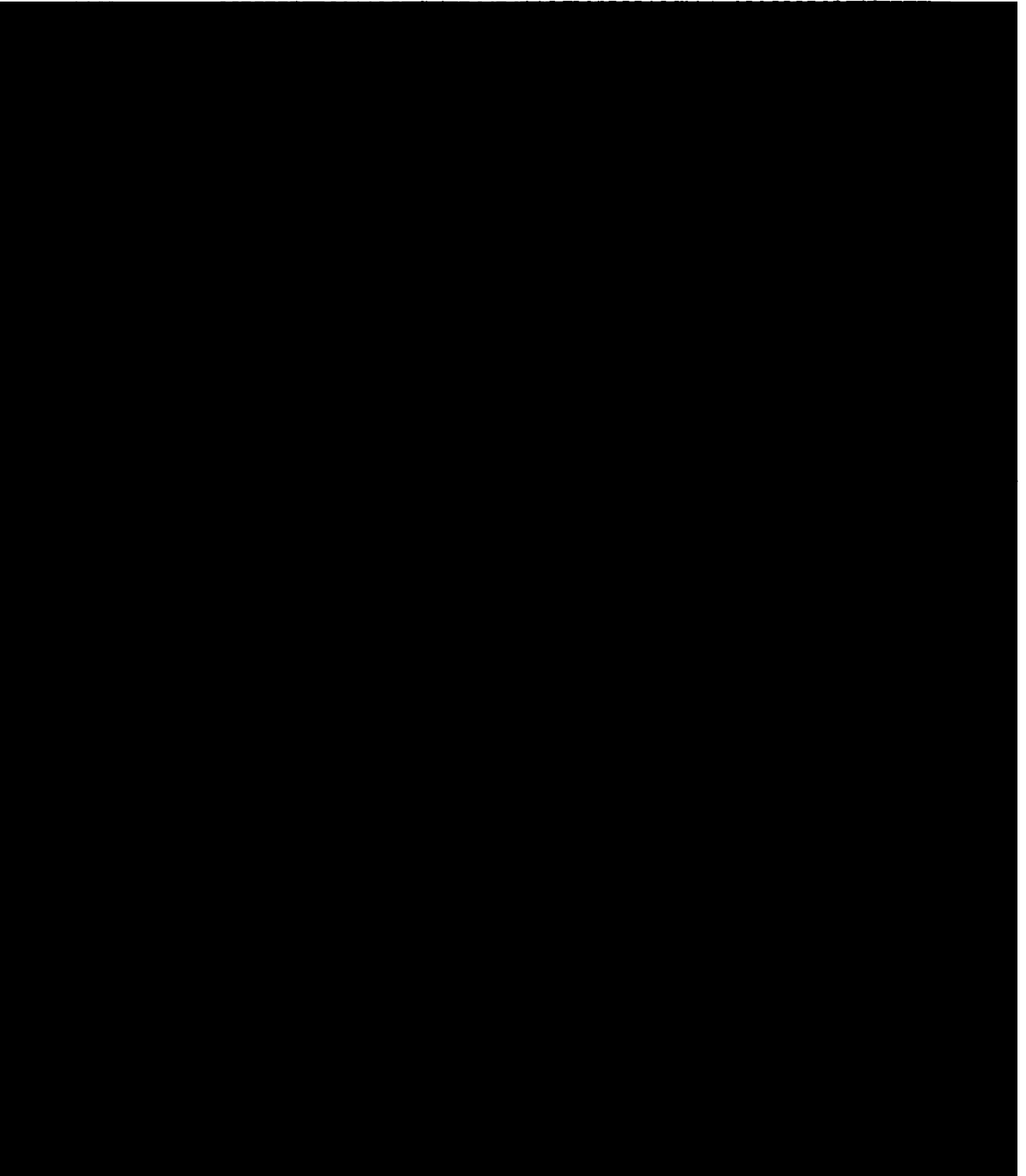


Figure 6-2 Location of Praire du Chien-Jordan Aquifer Wells

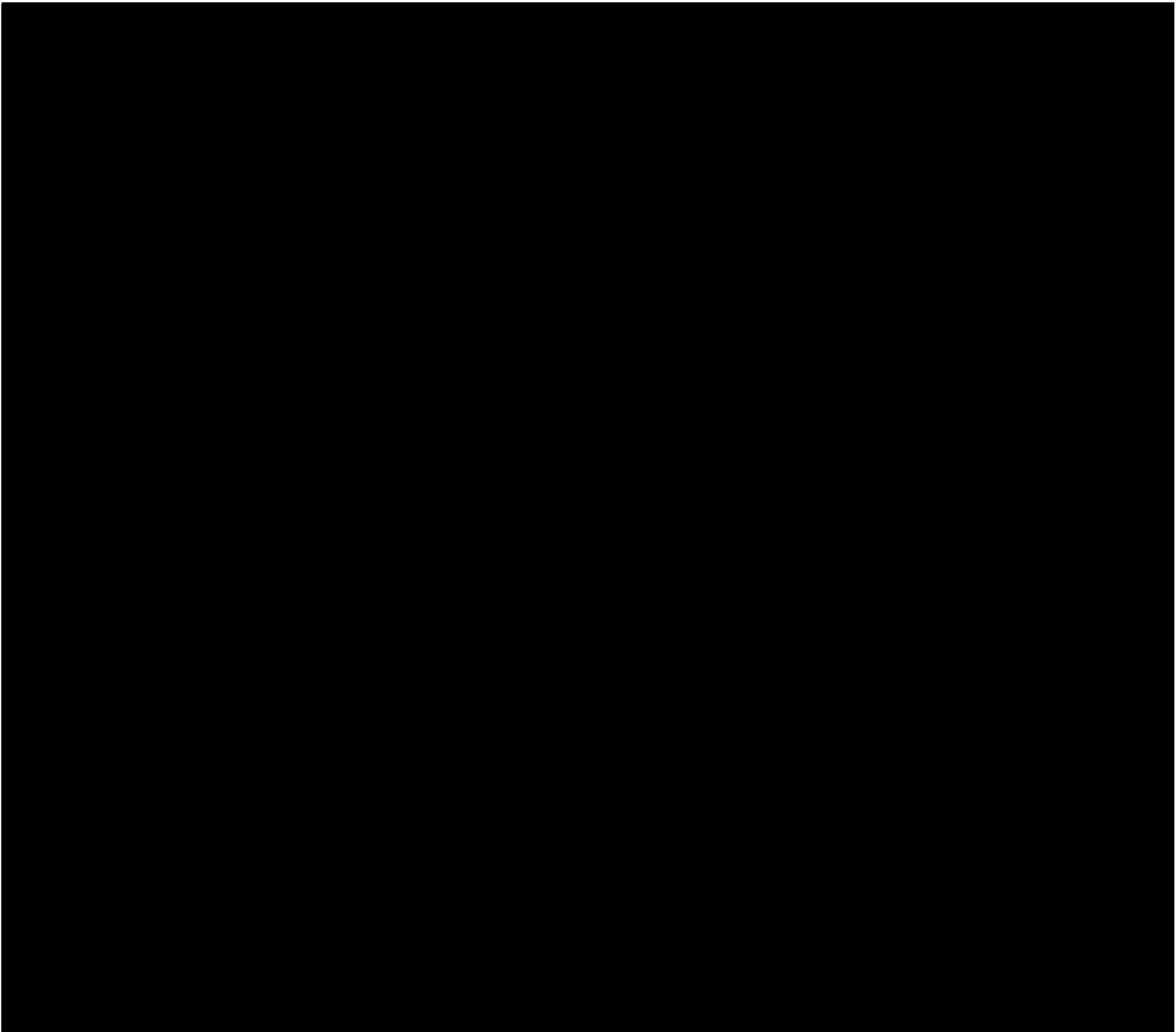


Figure 6-3 Location of Source and Gradient Control Wells

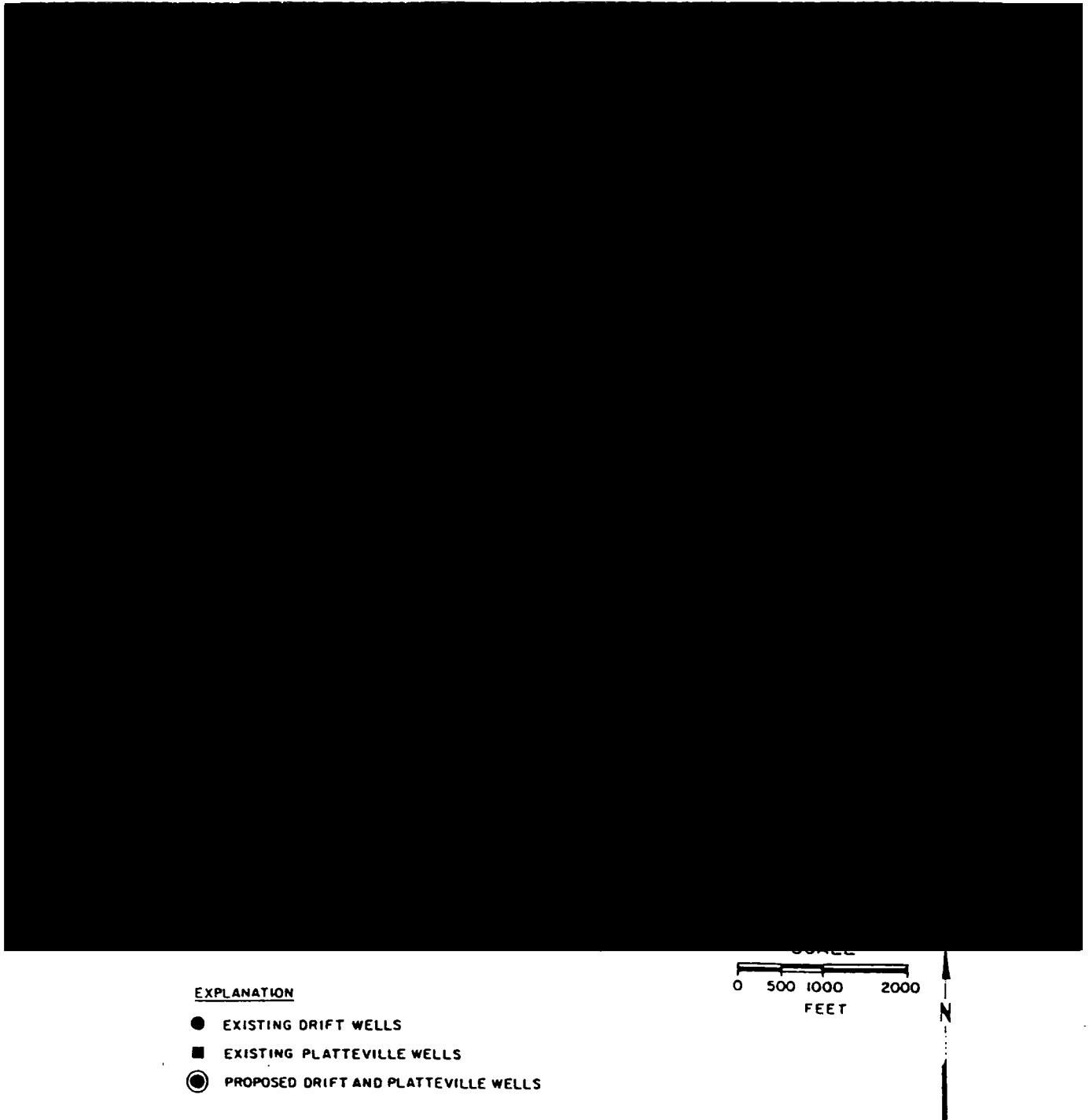
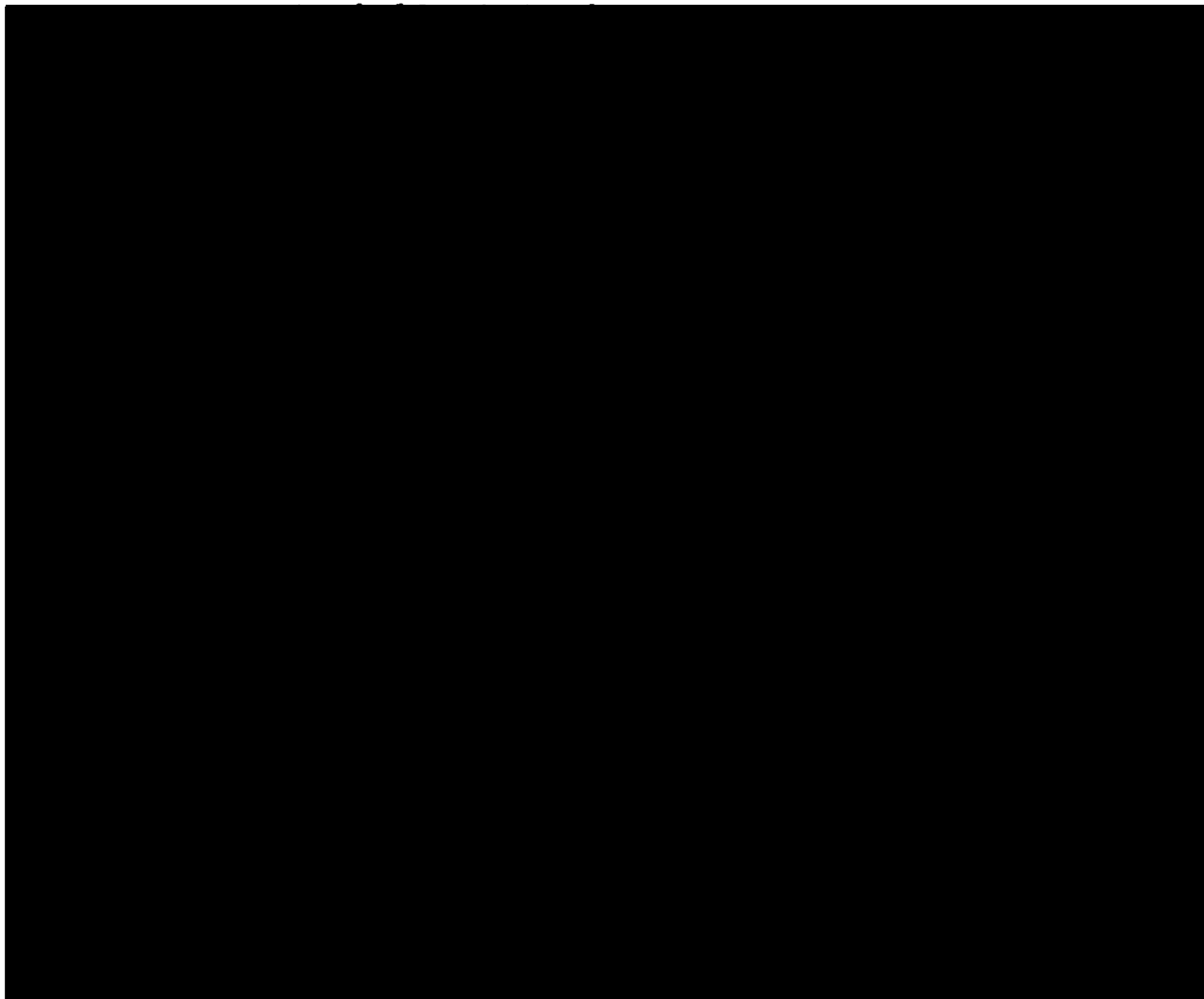


Figure 6-4 Location of Drift-Platteville Monitoring Wells

Reference: MGS, Miscellaneous Map Series,



EXPLANATION

- ▲ W 33 LOCATION AND PROJECT WELL NUMBER
- ▲ OBSERVATION WELL COMPLETED IN ST. PETER AQUIFER
- OBSERVATION WELL COMPLETED IN BASAL ST. PETER CONFINING BED
- NEW ST. PETER MONITORING WELLS
- ⊙ WELL IN WHICH WATER LEVELS WERE MONITORED WITH A DIGITAL RECORDER DURING PART OF 1978-81
- ▨ BEDROCK VALLEY/CONTACT WHERE UNCONSOLIDATED DRIFT DEPOSITS OVERLIE ST. PETER SANDSTONE

Figure 6-5 Proposed and Existing
St. Peter Aquifer Well Locations and Bedrock Valley

When any of these documents are revised, the affected pages are reissued to all personnel listed as document holders with updated revision numbers and dates. Issuance of revisions is accompanied by explicit instructions as to which documents or portions of documents have become obsolete.

Control of, and accounting for documents generated during the course of the project is achieved by assigning the responsibility for document issuance and archiving. Table 6-3 lists the key documentation media for the project and corresponding responsible parties for issuance, execution and archiving.

6.3 Sample Control Procedures and Chain of Custody

In addition to proper sample collection, preservation, storage and handling, appropriate sample identification procedures and chain of custody are necessary to help insure the validity of the data.

6.3.1 Sample Identification

Sample labels shall be completed for each sample, using waterproof ink, unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because a ballpoint pen would not function in freezing weather. The information recorded on the sample label includes:

Sample Number - Unique coded sample identification number as described below.

Time - A four-digit number indicating the military time of collection.

Sampler - Signature of person collecting the sample.

Remarks - Any pertinent observations or further sample description. The sample number includes three parts (source code, sampling point code, and date code) in the following sequence:

XXX-YYYYY-ZZZZZ

TABLE 6-3
 DOCUMENT CONTROL

<u>Item</u>	<u>Issued By</u>	<u>Issued To</u>	<u>Archived By</u>
Field Notebooks	Field Coordinator	Sampling Team	Field Coordinator
Field Equipment Calibration Forms	Field Coordinator	Sampling Team	Field Coordinator
Sample Logs	Field Coordinator	Sampling Team	Field Coordinator
Chain-of-Custody Forms	Lab Sample Custodian	Field Coordinator	Lab Sample Custodian
Sample Labels	Field Coordinator	Sampling Team	Lab Sample Custodian

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XXX = Source Code
GAC Plant = GAC
Mt. Simon-Hinckley Aquifer = MSH
Ironton-Galesville Aquifer = IGV
Prairie du Chien Jordan Aquifer = PCJ
St. Peter Aquifer = STP
Drift-Platteville Aquifer = DPV

YYYYY = Sampling Point Code
Well identification as abbreviated in Tables 6-1 and 6-2

ZZZZ = Date Code
Month, day, year

Those samples which will be taken in accordance with this Plan for quality control purposes will be identified by appending to the sampling point codes the following:

Field blank = FB
Field duplicate = D
Matrix spike = MS
Matrix spike duplicate = MSD

As an example, a field blank sample taken for the Mt. Simon-Hinckley Aquifer, sampling point SLP11 on 1 January 1988 would be identified as follows:

MSH-SLP11FB-010188

During the sampling event, one sample will be taken per sampling point unless it is duplicated. Duplicate samples will be collected as specified in Tables 6-1 and 6-2. Those samples collected for matrix spike analysis will be selected at the time of sampling and labelled in the field.

After collection, identification, and preservation, the sample will be maintained under chain-of-custody procedures discussed below.

6.3.2 Chain-of-Custody Procedures

To maintain and document sample possession, chain-of-custody procedures will be followed. A sample is under custody if:

- o It is in someone's possession, or
- o It is in someone's view, after being in their possession, or
- o It was in someone's possession and they locked it up to prevent tampering, or
- o It is in a designated secure area.

Samples are accompanied by a Chain-of-Custody Record (Figure 6-6). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst at the laboratory.

Minimum information recorded on the chain-of-custody record in addition to the signatures and dates of all custodians will include:

- o Sampling site identification
- o Sampling date and time
- o Identification of sample collector
- o Sample identification
- o Sample description (type and quantity)
- o Analyses to be performed.

Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. Shipping containers will be sealed for shipment to the laboratory. The method of shipment, courier name(s) and other pertinent information are entered in the "Remarks" box. Then tear off the last copy of the form and place the original and remaining copies in the container. After the container is closed, place the custody seals on the container.

Whenever samples are split with another laboratory, it is noted in the "Remarks" section. The note indicates with whom the samples are being split and is signed by both the sampler and recipient. If either party refuses a split sample, this will be noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

4955 Yarrow Street, Arvada, CO 80002 (303) 421-6611

**A DIVISION OF
ENSECO
INCORPORATED**

RMAL Client _____

RMAL Project No. _____

Sampling Co. _____

Sampling Personnel _____

Project Name/No. _____

Sampling Site _____

[illegible]

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time	Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Method of Shipment:		Shipped by: (Signature)		Delivered by: (Signature)		Received for Laboratory by: (Signature)	

White Copy to Lab

Pink Copy to Client

Yellow Copy to Sampler

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Figure 6-6 Sample Chain of Custody Record

6.3.3 Field Forms

In addition to sample labels and chain-of-custody forms, a bound field notebook will be maintained by the sample team leader to provide a daily record of significant events. All entries will be signed and dated. All members of the of the sampling team will use this notebook. The notebook will be kept as a permanent record.

6.4 Sampling Procedures - GAC Plant

Chain-of-custody forms will be completed and all samples shipped to RMAL's laboratory by overnight delivery on the same day they are collected.

Sampling points will be flushed for at least five minutes before collecting a sample. Each PAH sample will be collected in four one-liter amber glass bottles, which should be filled and capped in succession. PAH sample bottles will not be rinsed before being filled. The lids of all sample bottles will be taped using plastic adhesive tape after they are capped.

The GAC treated water samples will have to be collected from two sample taps -- one for each column (see Figure 6-7). This will be done by filling two one-liter bottles from the first column sample tap and then two more bottles from the second (four from each for duplicate samples). No notations distinguishing the two taps will be made on the labels. All four PAH bottles will be extracted and the extracts composited for analysis.

Field blank samples will be prepared by transferring contaminant-free deionized water provided by RMAL into sample bottles in a fashion as closely similar to actual sample collection as possible. Field blank sample bottles will be filled, capped and taped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area in which GAC treated water samples are collected.

Duplicate samples will be obtained by filling eight 1-liter bottles at the sampling point by the procedure described above, splitting these into two groups of four bottles, and assigning a different sample number to each of the resulting four-bottle samples. All samples will be packed, cooled to a temperature less than 4°C, and shipped on the day they are collected.

The sampling team must recognize that great care is required to collect samples for part-per-trillion-level PAH analysis that are free from outside contamination. PAH compounds are present in cigarette smoke, engine exhaust and many petroleum derived oils, among other sources. There will be no smoking anywhere in the GAC treatment building on a day on which PAH samples are to be collected until the samples have been collected, sealed and packaged for shipment. Similarly, no vehicles will enter the GAC treatment building and the large access door will stay closed on sampling days. Disposable gloves will be worn when collecting, handling and packaging samples. Sample bottles will remain in closed shipping coolers until they are needed, and will be packaged and sealed for shipment as soon as possible after sampling.

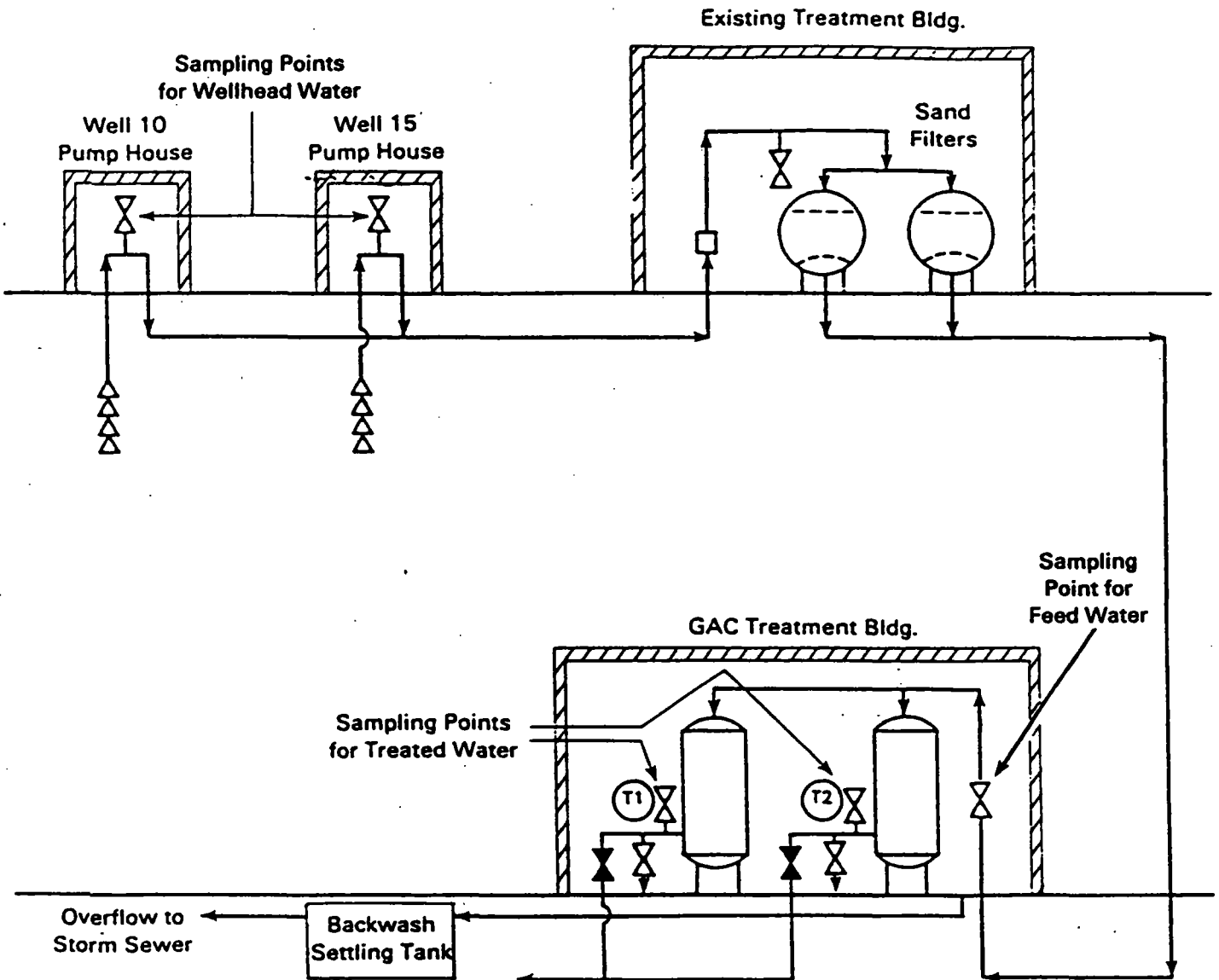


Figure 6-7 Sampling Locations

6.5 Ground-water Sampling and Water Level Measurements

Ground water samples will be collected and water level measured in accordance with the procedures outlined in this Plan. The wells involved in the monitoring program include municipal and commercial wells, piezometers and groundwater monitoring wells (see Table 6-2). Sampling procedures to accommodate the dimensions and configuration of each type of well are described below. Further details on well dimensions, water level measurements and sample acquisition strategies are given in the Site Management Plan.

The importance of proper sampling of wells cannot be over-emphasized. Even though the well being sampled may be correctly located and constructed, special precautions must be taken to ensure that the sample taken from that well is representative of the ground water at that location and that the sample is neither altered nor contaminated by the sampling and handling procedure. Sample collection will always proceed from the less contaminated sampling points to the monitoring wells containing progressively higher concentrations of PAH or phenolics.

6.5.1 Decontamination

The field decontamination procedure to be used on sampling equipment which comes into contact with groundwater samples is as follows:

- o disassemble equipment, if applicable,
- o high pressure, hot water steam clean, using potable water.

The laboratory decontamination procedure to be used on sampling equipment which comes into contact with groundwater samples is as follows:

- o disassemble equipment
- o rinse with acetone
- o scrub with hot soapy water
- o rinse three times with hot deionized water
- o set on aluminum foil, dull side up, air dry
- o bake for one hour at 200° C
- o wrap with aluminum foil, dull side in

6.5.2 Field Blanks

Field blank samples will be prepared by transferring contaminant-free deionized water, provided by RMAL, into sample bottles in a fashion as closely similar to actual sample collection as possible. This will involve collecting samples through any non-dedicated sample equipment that is decontaminated between samples. Field blank sample bottles will be filled, capped and taped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area where samples are being collected at a rate of one per day or where more than ten samples are collected in a day at a rate of one field blank per ten samples.

6.5.3 Sample Containers (See Table 6-4)

For PAH and Phenolics, 1 liter amber glass bottles will be used. Caps will be fitted with pre-cleaned Teflon liners. Four bottles are required for each PAH sample collected. One bottle is required for phenolics.

Bottles will be prepared as follows:

1. Wash bottles with hot detergent water.
2. Rinse thoroughly with tap water followed by three or more rinses with organic-free water.
3. Rinse with Burdick & Jackson quality redistilled acetone, followed by equivalent quality methylene chloride.
4. Allow to air dry in a contaminant free area.
5. Caps and liners must be washed and rinsed also.

Bottles should be stored and shipped with the Teflon-lined caps securely fastened.

6.5.4 Sample Collection - Monitoring Wells and Piezometers

Because unanticipated or changed conditions may cause difficulty in the purging and sampling of the monitoring wells and piezometers, flexibility in the approach to sample retrieval is necessary. This Plan proposes that the sampling team be given latitude in the selection of purge/sample equipment and procedures necessary to complete the monitoring task.

Table 6-2 specifies that Prairie du Chien-Jordan Aquifer monitor well W70, St. Peter Aquifer monitor wells W24 and W33 and Drift-Platteville Aquifer monitor well W117 be monitored. Each well is equipped with a dedicated submersible pump and it will be the responsibility of the sampling team to determine if the pump is operable. In the event the dedicated pump within any individual well is operable, well purging and sample retrieval tasks will be completed with the aid of the pump in conformance with parameter monitoring established herein. In the event the dedicated pump within any individual well is inoperable, the pump will be removed and purging/sampling procedures will be as established below.

Monitoring wells and piezometers not equipped with dedicated submersible pumps will be purged using a nondedicated submersible pump, suction pump or bailer. During the purging of each well, temperature, pH and specific conductance of the purge water will be monitored using a Hydrolab water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of these readings will indicate that purging is complete and sampling may

TABLE 6-4
SAMPLE CONTAINERS, PRESERVATION PROCEDURES, AND
MAXIMUM HOLDING TIMES

<u>Parameter</u>	<u>Containers</u>	<u>Preservation¹</u>	<u>Maximum Holding Time²</u>
Water; PAH (PPT)	Four 1-liter amber glass bottles, Teflon-lined caps	cool, to 4° C; protect from light	7 days (until extraction), 40 days after extraction
PAH (PPB)	Two 1-liter amber glass bottles, Teflon-lined caps	cool, to 4° C; protect from light	7 days (until extraction), 40 days after extraction
Phenolics	One 1-liter amber glass bottle,	cool, to 4° C	7 days (until extraction), 40 days after extraction

Ref: Federal Register Guidelines/Vol.49, No.209/Friday, October 26, 1984/p. 43260.

- 1 Sample preservation will be performed immediately upon sample collection.
- 2 Samples will be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

commence. Upon completion of well purging, samples will be collected from each well using a stainless steel or teflon bailer and a new length of nylon or polyester rope. All nondedicated purging and sampling equipment will be decontaminated before use and between sampling points as described in Section 6.5.1.

Samples will be collected by filling each of the appropriate sample containers in rapid succession, without prerinsing the containers with sample. The bottle will be held under the sample stream without allowing the mouth of the bottle to come in contact with the bailer and filled completely, and the cap securely tightened. Bottles will be checked for air and if air is visible, the cap removed and more sample added. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

6.5.5 Sample Collection - Pumping Wells

At active pumping wells the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team.

Water level measurements will then be made, if practical. The normal operation of the well will not be interrupted for the purpose of measuring water levels. An electric tape will be used to measure water levels in pumping wells. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered.

If it can not be determined that a well has been pumping at some time during the 24 hour period preceding sampling, or if it is known the well was not pumping, then the well shall be purged until field measurements of temperature, pH, and specific conductance have stabilized after at least three well volumes have been removed from the well. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

6.6 Sample Preservation, Shipment and Storage

The samples will be iced or refrigerated at 4°C from the time of collection until extraction. PAH's are known to be light sensitive; therefore, samples will be stored in amber bottles and kept away from prolonged exposure to light. All samples will be extracted within seven days of collection, and analysis completed within forty days following extraction.

Samples will be protected from breakage and shipped in coolers at a temperature of 4°C or less. An overnight carrier will be selected to insure delivery at the laboratory within 24-36 hours after collection.

Samples received at the laboratory will be checked for leakage and a notation made regarding sample temperature at time of receipt. All samples should be stored in an organic-free refrigerator at 4°C. Storage refrigerators will be kept locked to prevent unauthorized entry and to satisfy chain-of-custody requirements.

6.7 Field Measurement Equipment

All field measurement equipment will be controlled to ensure that measurements obtained are accurate and defensible. Table 6-5 summarizes the parameters to be monitored, the instruments to be used for each measurement, procedures including calibration and frequency, and quality control criteria (also refer to Appendix A, SOP 7320, Calibration and Operation of Hydrolab Water Quality Monitor).

In addition, these measurement devices will be issued through a formal equipment tracking system and operated by trained personnel.

6.8 Duplicate Samples

Duplicate samples will be collected by alternately filling sample bottles from the source being sampled. For four liter sample collection one bottle will be filled for the sample, then one bottle for the duplicate, then a second bottle for the sample and then a second bottle for the duplicate, etc. Duplicates will be taken for each analysis type and each sample type, at a rate of one duplicate sample being collected for each ten samples, with a minimum of one duplicate for any sample batch. There are two sample types for this program: GAC Plant treated water and groundwater. For purposes of fulfilling the 10% duplicate requirement, all the sampling points shown on Table 6-2 are the same sample type.

TABLE 6-5
FIELD MEASUREMENT EQUIPMENT QUALITY CONTROL

<u>Device</u>	<u>Calibration</u>	<u>Routine Check</u>		<u>Control Limits</u>
		<u>Method</u>	<u>Frequency</u>	
pH Meter (Hydrolab)	Standardize in three or more standard buffer solutions	Calibration check-analyze standard buffer solution	after every sample	± 0.1 pH units
		Analyze duplicates	after every sample	± 0.1 pH units
Conductivity Meter (Hydrolab)	Standardize using two or more KCL solutions	Calibration check-analyze standard KCL solution	1/10 Samples	$\pm 10\%$ full scale
		Analyze duplicates	1/10 Samples	$\pm 10\%$ full scale

7. SAMPLE CUSTODY

The St. Louis Park Groundwater Study is a cooperative effort between the City and ERT, whose responsibilities include sample retrieval, and RMAL, whose responsibilities include sample analysis. Proper sample handling and analysis is essential to the success of the study, therefore a formal sample custody procedure has been developed to insure the integrity of all samples. Sections 6.4 and 6.5 discuss field sampling aspects and Section 6.6 outlines procedures for sample preservation, shipment, and storage. This section covers quality related activities from receipt of samples at the RMAL analytical facilities through issuance of validated analytical data and the storage of data in the final evidence file.

7.1 Security and Recordkeeping

Samples entering the RMAL analytical facilities located in Arvada, Colorado, proceed through an orderly chain-of-custody sequence specifically designed to insure continuous integrity of both the sample and documentation.

Appendix A contains Standard Operating Procedures (SOP's) which address the following aspects of facility security and sample custody

- o Building Security - SOP No. LP-RMA-0001
- o Sample Receipt and Chain of Custody - SOP No. LP-RMA-0005
- o Project Assignment Record - LP-RMA-0004
- o Sample Log-in Procedures - LP-RMA-0003

7.2 Final Evidence File

The final evidence (or data) files from RMAL will be maintained by the City for the period specified in the RAP. Evidence files will consist of all data necessary to completely reconstruct the analysis, and will consist of (at a minimum): raw data, continuing calibration checks, DFTPP tune, detection limits, chain of custody documentation, quality control data for blanks and matrix spikes and results forms. In addition, the analytical report, which contains a brief discussion of the method and a more detailed narrative of any analytical issues is included in the package. The City will maintain these files in a secure, limited access area under the custody of the Director of Public Works or the City Manager. RMAL maintains all GC/MS raw data files on tapes or other magnetic media for an indefinite period. This data will be available upon request.

8. CALIBRATION PROCEDURES

8.1 Low-Level (ppt) Analysis of PAH and Heterocycles

Properly calibrated instrumentation is required for accurate quantitation of PAH and heterocycles. To accomplish this, a number of specific procedures have been instituted in the laboratory. Prior to use of the method for low level analysis of PAH and Heterocycles, a five-point response factor calibration curve must be established showing the linear range of the analysis. For every 12 hours of GC/MS analysis, the mass spectrometer response for each PAH or heterocycle relative to the internal standard is determined, using daily check standards at concentrations of 40 ng/mL. Daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within ± 35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within ± 35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

Chromatographic peak location criteria will be established using relative retention time. Relative retention times of daily check standards must be within the 95 percent confidence limits calculated from the calibration standards for each PAH or heterocyclic compound. In addition, sample component relative retention times must be within ± 0.1 relative retention time units of the standard component relative retention time. Similar procedures will be followed for the extended analysis for carcinogenic PAH in the GAC Plant and non-criterion PAH analyses.

8.2 Total Phenols

Calibration for the analysis of total phenols (phenolics) will be accomplished through the selection of appropriate standards. A calibration curve will be established to which the working calibration curve will be verified daily.

9. ANALYTICAL PROCEDURES

9.1 Low Level Analysis of PAH and Heterocycles

A method has been developed for the analysis of selected target PAH and heterocycle compounds at the part per trillion level (ppt, ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by gas chromatography mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). The compounds listed in Table 9-1 can be quantitatively determined using this analytical method.

Four 1-liter volumes of sample are separated into two 2-liter samples and extracted with methylene chloride. Analysis of the combined and concentrated extract is performed by gas chromatography/mass spectrometry using the selected ion monitoring scanning mode under electron impact ionization conditions. Specific details of this methodology can be found in Appendix B, Determination of Low Level (Part Per Trillion) PAH and Heterocycles in Water.

9.2 Extended Analyses for Carcinogenic PAH in GAC Plant

To satisfy the requirements of the RAP Section 4.3.4, RMAL will analyze one sample per year of the GAC treated water for the additional carcinogenic compounds shown on Table 9-2 and search for additional compounds that may be present. RMAL will first analyze the sample according to the low level PAH analytical methodology. A calibration standard containing the compounds shown on Table 9-3 will be prepared and used to establish a five point calibration curve. All calibration procedures outlined in Appendix B, Determination of Low Level (Part per Trillion) PAH and Heterocycles in Water will be followed.

The sample will be extracted, prepared and analyzed as outlined in the low level PAH analytical methodology, generating quantitative results for the compounds being regularly measured. A second injection will be made with a selective ion monitoring program using the quantitation masses shown in Table 9-2. This will allow the extended analysis compounds to be quantitated at an approximately 2 ppt detection limit.

Following the quantitative analyses of the regular and extended analysis compounds, the extract will be reduced to a 50 ul final volume. An aliquot will be analyzed using full-scan GC/MS (40-500 amu). Any peaks having a signal to noise ratio of 5 or larger will be identified, if possible, using the EPA/NIH mass spectral library. Compounds so identified will be quantitated using the nearest internal standard and a response factor of 1.0, to a detection limit of approximately 5 ppt.

9.3 Analyses for Phenolics

Total phenolics analyses will be performed using EPA Method 420.2. The complete methodology is contained in Appendix B, Phenolics, Total Recoverable, Method 420.2 (colorimetric, automated 4-AAP with distillation).

9.3.1 Extended Analysis for Phenolics in GAC Plant

To satisfy the requirements of the RAP section 4.3.4, RMAL will analyze one sample per year of GAC treated water for the acid fraction compounds in EPA Test Method 625. The CLP protocol (Section IV, Exhibit D, SOW 7/87) will be used for this analysis.

9.4 Expanded Analyses

If expanded analyses are required in accordance with RAP Section 9.3.3, an addendum will be written to the QAPP to encompass these analyses.

9.5 Non-Criteria PAH Analyses

Non-criteria PAH samples will be analyzed, according to CLP Protocol, Exhibits D and E, Section IV, Exhibit D, SOW 7/87 (see Appendix B) with the following exceptions:

- 1) The compounds analyzed list will be limited to those compounds listed in QAPP Table 9-1.
- 2) Deuterated PAH will be used for surrogates and internal standards, as shown on Table 9-1.
- 3) Matrix spikes will be analyzed as detailed in QAPP Section 11.1.4 using the select list of matrix spike compounds as shown therein.
- 4) Surrogate and matrix spike acceptance criteria will be those given in QAPP Section 15.1.

As described in the method, a one-liter water sample will be extracted and analyzed, to give a reported detection limit of 10 parts per billion for each compound.

TABLE 9-1
COMPOUNDS AND MS QUANTITATION MASS IONS

<u>Compound</u>	<u>Quantitation Mass Ion</u>	<u>Confirmation Ion (% Abundance)</u>	<u>Internal Standard Reference</u>
<u>Polynuclear Aromatic Hydrocarbons (PAH)</u>			
Naphthalene	128	102 (7)*	1
Acenaphthylene	152	151 (17)*	1
Acenaphthene	154	153 (93)*	1
Fluorene	166	165 (90)*	1
Phenanthrene	178	176 (19)*	2
Anthracene	178	176 (19)*	2
Fluoranthene	202	200 (17)*	2
Pyrene	202	200 (18)*	2
Benzo(a)anthracene	228	226 (22)*	3
Chrysene	228	226 (26)*	3
Benzo(a)pyrene	252	250 (22)*	3
Indeno(1,2,3,cd)pyrene	276	274 (21)*	3
Dibenz(a,h)anthracene	278	279 (20)*	3
Benzo(g,h,i)perylene	276	274 (25)*	3

Internal Standards

1) Acenaphthene-d10	164	--
2) Phenanthrene-d10	188	--
3) Benzo(a)pyrene-d12	264	--

Surrogates

1) Naphthalene-d8	136	1
2) Fluorene-d10	176	1
3) Chrysene-d12	240	3

*The % abundance for the confirmation ion is a typical value obtained during the method detection limit study. Although these ratios will vary, the relative intensities of confirmation ions must agree within plus or minus 20% between the calibration standard for any given day and the samples run on that day.

TABLE 9-1 (Continued)
COMPOUNDS AND MS QUANTITATION MASS IONS

<u>Compound</u>	<u>Quantitation Mass Ion</u>	<u>Confirmation Ion (% Abundance)</u>	<u>Internal Standard Reference</u>
<u>Heterocycles and Other PAH</u>			
Indene	116	115 (108)*	1
Indole	117	90 (52)*	1
2,3-dihydroindene	117	118 (57)*	1
2,3-benzofuran	118	90 (31)*	1
Quinoline	129	102 (20)*	1
Benzo(b)thiophene	134	89 (8)*	1
2-methylnaphthalene	141	115 (31)*	1
1-methylnaphthalene	141	115 (28)*	1
Biphenyl	154	153 (35)*	1
Carbazole	167	166 (28)*	2
Dibenzofuran	168	139 (40)*	1
Acridine	179	178 (26)*	2
Dibenzothiophene	184	139 (19)*	2
Perylene	252	250 (24)*	3
Benzo(e)pyrene	252	250 (35)*	3

Internal Standards

1) Acenaphthene-d10	164	--
2) Phenanthrene-d10	188	--
3) Benzo(a)pyrene-d12	264	--

Surrogates

1) Naphthalene-d8	136	1
2) Fluorene-d10	176	2
3) Chrysene-d12	240	3

*The % abundance for the confirmation ion is a typical value obtained during the method detection limit study. Although these ratios will vary, the relative intensities of confirmation ions must agree within plus or minus 20% between the calibration standard for any given day and the samples run on that day.

TABLE 9-2
EXTENDED ANALYSIS CARCINOGENIC PAH

<u>Compound</u>	<u>Quantitation Mass</u>
benzo(c)phenanthrene	226
dibenz(a,c)anthracene	278
dibenzo(a,e)pyrene	276
dibenzo(a,h)pyrene	276
dibenzo(a,i)pyrene	276
7,12-dimethylbenz(a)anthracene	256
3-methylcholanthrene	268

TABLE 9-3
RELATIVE RETENTION TIMES AND CONFIDENCE LIMITS FOR THE COMPOUNDS
ASSOCIATED WITH THE LOW LEVEL PAH AND HETEROCYCLE METHODOLOGY

	<u>Absolute Retention Time (minutes)</u>	<u>Avg. RRT</u>	<u>SD</u>	<u>% RSD</u>	<u>95% Confidence Limits</u>
Benzofuran	8:03	0.550	0.015	2.807	0.520-0.580
Dihydroindene	8:45	0.590	0.016	2.765	0.558-0.622
Indene	8:54	0.598	0.016	2.699	0.566-0.630
Naphthalene-d8(Surr.)	11:14	0.733	0.017	2.289	0.699-0.767
Naphthalene	11:16	0.735	0.017	2.289	0.701-0.769
Benzo(b)thiophene	11:25	0.743	0.017	2.258	0.709-0.777
Quinoline	12:06	0.783	0.017	2.140	0.749-0.817
Indole	12:55	0.824	0.018	2.167	0.788-0.860
2-methylnaphthalene	12:59	0.832	0.017	2.084	0.798-0.866
1-methylnaphthalene	13:15	0.848	0.017	2.055	0.814-0.882
Biphenyl	14:12	0.901	0.017	1.921	0.867-0.935
Acenaphthylene	15:15	0.962	0.018	1.822	0.927-0.988
Acenaphthene	15:44	0.988	0.018	1.849	0.952-1.024
Dibenzofuran	16:09	1.011	0.018	1.791	0.975-1.047
Fluorene-d10(Surr.)	16:57	0.872	0.015	1.735	0.842-0.902
Fluorene	17:01	0.875	0.015	1.745	0.845-0.905
Dibenzothiophene	19:08	0.974	0.016	1.617	0.942-1.006
Phenanthrene	19:28	0.988	0.016	1.589	0.956-1.020
Anthracene	19:34	0.994	0.016	1.597	0.962-1.026
Acridine	19:42	0.999	0.016	1.572	0.967-1.031
Carbazole	20:02	1.013	0.015	1.487	0.983-1.043
Fluoranthene	22:32	1.130	0.017	1.461	1.096-1.164
Pyrene	23:07	1.157	0.017	1.443	1.123-1.191
Benz(a)anthracene	26:16	0.873	0.012	1.325	0.849-0.897
Chrysene-d12 (Surr.)	26:18	0.874	0.012	1.320	0.850-0.898
Chrysene	26:22	0.876	0.012	1.320	0.852-0.900
Benzofluoranthenes	29:00	0.960	0.014	1.501	0.932-0.988
Benzo(e)pyrene	29:34	0.984	0.016	1.590	0.952-1.016
Benzo(a)pyrene	29:44	0.988	0.016	1.615	0.956-1.020
Perylene	29:55	0.996	0.016	1.644	0.964-1.028
Indeno(1,2,3 cd)pyrene	32:31	1.114	0.025	2.276	1.064-1.164
Dibenz(ah)anthracene	32:36	1.113	0.031	2.743	1.051-1.175
Benzo(ghi)perylene	33:17	1.149	0.028	2.422	1.093-1.205

10. DATA REDUCTION, VALIDATION AND REPORTING

10.1 Data Reduction and Validation

All project data will be subjected to a three-tier review process including review by operations, the data review group for inorganics, GC/MS, and chromatography and the final review by the project or client managers prior to its release to the client. The review process has been developed to minimize errors associated with sample processing, sample analysis and data reporting and to ensure that information pertaining to a given sample is well documented.

Appendix A contains Standard Operating Procedures (SOP's) for laboratory data review. Refer to SOP No. LP-RMA-0002 for information relative to review policies and processes.

10.2 Turnaround Time

In accordance with Section 3.2 of the RAP, RMAL has agreed to a 30 working day turnaround. The City, however, makes no enforceable commitment under the RAP except for a maximum of 7 days from sampling for extraction of organics and 40 days following extraction for analysis of organics. For non-organic analyses, the City makes no enforceable commitment under the RAP except to meet the recommended maximum analytical holding times.

10.3 Reporting/Data Deliverables

RMAL shall submit reports and data packages to the City in a format described in Exhibit B of Organic SOW 7/87 for the Contract Lab Program. The reports and data packages will be compiled by the City.

The various items in the data package are listed below:

- o Sample Traffic Reports or Chain-of-Custody
- o Sample Data Summary Package Including:
 - Case narrative
 - Tabulated target compound results by fraction
 - Surrogate spike analysis results by fraction
 - Matrix spike/matrix spike duplicate results by fraction
 - Blank data by fraction
- o Sample Data Package including:
 - Case narrative
 - Traffic reports
 - Volatiles Data
 - Semivolatiles Data
 - Pesticide Data

The volatiles, semivolatiles and pesticides data packages will include a QC summary, the raw sample data, standards data and raw QC data.

The City will present reports in a manner consistent with the requirements of the RAP. In addition, data packages containing all elements listed above will be presented for up to 10 percent of the sample analyses completed. The EPA shall be responsible for identifying the specific sample analyses for which data packages will be provided.

10.4 Reporting Requirements for Samples Exceeding Advisory Levels or Drinking Water Criterion

For active drinking water wells, RMAL will notify the City of St. Louis Park by telephone, within 24 hours of completing an analysis, whenever a sample analysis is shown to exceed the following Advisory Levels or Drinking Water Criterion:

<u>Parameter</u>	<u>Advisory Level</u>	<u>Drinking Water Criterion</u>
Sum of Benzo(a)pyrene and Dibenz(a,h)anthracene*	3.0 ng/L*	5.6 ng/L
Total Carcinogenic PAH	15 ng/L**	28 ng/L**
Total Other PAH	175 ng/L	280 ng/L

*Or the detection limit, whichever is largest.

**Different concentrations for additional carcinogenic PAH may be established in accordance with the procedure specified in Part D.1 of the Consent Decree.

10.5 Final Evidence Files

The final evidence (or data) files from RMAL will be maintained by the City for the period specified in the RAP. Evidence files will consist of all data necessary to completely reconstruct the analysis, and will consist of, (at a minimum): raw data, calibrations, QC, detection limits, result forms and the analytical report. The City will maintain these files in a secure, limited access area under the custody of the Director of Public Works or the City Manager.

11. INTERNAL QUALITY CONTROL CHECK**11.1 Low-level PAH Analyses/Extended Analyses/Non-Criteria PAH Analyses**

A program ensuring proper laboratory operation includes a number of types of methodological checks. These include detection limit studies, method and solvent blank analyses, surrogate analyses, matrix spike analyses and duplicate analyses. The following quality control checks have been developed specifically for the Reilly Tar and Chemical Corporation N.P.L. site, St. Louis Park, Minnesota.

11.1.1 Method Detection Limit

RMAL has determined the method detection limits for the part per trillion PAH analysis of water samples, utilizing GC/MS selected ion monitoring, according to the method described in Appendix B to Part 136 of the Friday, October 26, 1984 Federal Register, Vol. 49, No. 209 - Definition and Procedure for the Determination of the Method Detection Limit - Revision 11.1. Table 11-1 lists the compounds, the observed concentrations of seven replicates spiked at 5 parts per trillion, the standard deviations and the method detection limits.

These calculated method detection limits will be used in sample reporting as follows:

- o Analytes detected at concentrations greater than or equal to the calculated method detection limits will be reported with no qualifiers.
- o Analytes that are detected at concentrations less than the calculated method detection limits will be reported followed by a "J" qualifier which is used in the EPA Contract Lab Program (CLP) to indicate that a reported value is below the method detection limit.

11.1.2 Method Blank

The laboratory will analyze method blanks according to CLP protocol. A method blank analysis must be performed once:

- o each Case, OR
- o each 14 calendar day period during which samples in a Case are received (said period beginning with the receipt of the first sample in that Sample Delivery Group), OR
- o each 20 samples in a Case that are of similar matrix (water or soil) or similar concentration (soil only), OR

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Compound	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Sample #7	Standard Deviation (s)	Method Detection Limit (3s)
2,3-Benzofuran	19.4*	20.9*	18.8*	19.5*	20.3*	21.5*	16.6*	1.70*	5.1*
2,3-Dihydroindene	4.3	4.2	4.7	3.7	3.8	4.9	4.7	0.48	1.4
1H-Indene	4.4	4.2	4.6	3.9	4.1	4.7	4.6	0.38	0.9
Naphthalene	20.5*	21.6*	18.5*	20.3*	23.0*	23.5*	17.6*	2.15*	0.5*
Benzo(B)thiophene	3.6	3.5	3.9	3.4	3.3	3.8	4.1	0.29	0.9
Quinoline	3.7	4.8	4.1	3.7	3.3	4.4	4.1	0.46	1.4
1H-Indole	4.7	4.5	5.0	3.2	3.2	4.2	4.8	0.84	2.5
2-Methylnaphthalene	5.4	5.0	5.3	5.1	4.8	4.9	5.7	0.31	0.9
1-Methylnaphthalene	4.5	4.2	4.6	3.8	3.7	4.7	5.2	0.53	1.6
Biphenyl	17.9*	18.1*	16.4*	18.4*	18.1*	19.3*	15.8*	1.43*	4.3*
Acenaphthylene	3.9	3.6	4.6	3.7	3.5	4.4	4.5	0.46	1.4
Acenaphthene	4.2	3.7	4.7	3.5	3.5	4.1	4.1	0.43	1.3
Dibenzofuran	4.3	3.9	4.6	4.1	3.7	4.6	4.2	0.34	1.0
Fluorene	4.4	4.6	4.5	4.6	4.6	4.6	4.8	0.33	1.0
Dibenzothiofene	4.0	3.5	4.0	3.5	3.2	3.9	4.2	0.36	1.1
Phenanthrene	4.7	3.9	4.7	3.9	3.6	4.2	4.5	0.43	1.3
Anthracene	4.5	3.8	4.5	4.1	3.6	4.1	4.6	0.38	1.1
Acridine	4.1	4.3	4.9	4.1	3.8	2.4	2.3	0.88	2.9
Carbazole	4.5	3.2	4.0	3.5	3.9	3.1	3.8	0.64	1.9
Fluoranthene	4.5	3.8	4.7	3.9	3.6	4.4	4.7	0.45	1.4
Pyrene	4.3	3.7	4.4	3.9	3.4	4.2	4.7	0.45	1.4
Benzo(A)anthracene	4.6	3.6	4.6	3.6	3.3	5.3	5.3	0.83	2.5
Chrysene	4.3	3.3	3.7	3.3	2.9	5.1	5.3	0.94	2.8
Benzo(B)fluoranthrene	4.6	3.4	3.8	3.6	2.8	4.9	5.0	0.83	2.5
Benzo(K)fluoranthrene	4.1	3.2	3.5	3.2	3.2	4.9	4.8	0.76	2.3
7,12-Dimethylbenzanthracene	5.3	3.9	5.5	5.3	4.3	6.6	6.6	0.93	2.8
Benzo(E)pyrene	4.9	3.8	4.1	3.3	3.5	4.9	4.4	0.64	1.9
Benzo(A)pyrene	4.5	3.2	3.8	3.2	2.9	4.8	4.5	0.76	2.3
Perylene	4.6	3.6	3.8	3.5	3.3	5.3	5.1	0.82	2.5
3-Methylcholanthrene	4.3	4.1	3.9	3.4	3.2	4.9	6.7	1.18	3.5
Indeno(1,2,3-CD)pyrene	4.5	3.4	3.4	2.9	3.6	4.5	4.2	0.69	2.1
Dibenz(A,C)anthracene **	4.2	3.5	3.6	3.1	3.3	4.6	4.1	0.54	1.6
Dibenz(A,H)anthracene **	4.2	3.5	3.6	3.1	3.3	4.6	4.1	0.54	1.6
Benzo(G,H,I)perylene	3.8	3.6	2.9	2.6	2.9	4.9	4.7	0.94	2.8

Note: Amount spiked = 5 ng/L.

* Data for 2,3-Benzofuran, Naphthalene and Biphenyl were obtained from previous detection limit study. Spike levels = 20 ng/L.

** Compounds co-elute

TABLE 11-1
METHOD DETECTION LIMIT REPORT

- o whenever samples are extracted by the same procedure (separatory funnel, continuous liquid-liquid extraction, or sonication),
whichever is more frequent, on each GC/MS or GC system used to analyze samples.

The method blank results associated with the sample batch will not be used to correct the observed sample concentrations in that batch. If an analyte is found in the method blank as well as in the sample, a "B" data qualifier will be added to the analyte reported in the analytical results table. This will indicate to the user the possibility of blank contamination.

11.1.3 Surrogates

The laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compounds will be spiked into the sample prior to extraction and, thus, will measure individual sample matrix effects associated with sample preparation and analysis. They will include naphthalene-d₈, fluorene-d₁₀ and chrysene-d₁₂, at a sample concentration level of 10 ng/L (ppt) or 20 ug/L (ppb).

A sample will be invalid for quantitative use in this program only if the recovery of any one or more of the surrogates falls outside the acceptance criteria. The initial acceptance criteria used for this program are the criteria established by ERT for these surrogates during 1986. The surrogate recovery acceptance criteria will be updated quarterly. RMAL will take corrective action whenever the surrogate recovery for any one or more surrogates is outside the following acceptance criteria:

<u>Surrogate</u>	<u>Acceptance Criteria %</u>	
	<u>Low-level</u>	<u>Non-criteria</u>
Naphthalene-d ₈	14-108	25-175
Fluorene-d ₁₀	41-162	25-175
Chrysene-d ₁₂	10-118	25-175

The following corrective action will be taken when required as stated above:

- a) Check calculations to assure there are no errors;
- b) Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
- c) Reanalyze the sample or extract if the steps in part a) or b) fail to reveal a problem. If reanalysis of the extracts yields surrogate spike recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported.
- d) If a), b) or c) do not correct the problem, the data for that sample will be reported but will not count towards satisfying the monitoring requirements of the RAP.

11.1.4 Matrix Spikes

The laboratory will spike and analyze 5% matrix spike samples. RMAL will spike seven representative compounds into water from the GAC plant or other well for ppt analyses and field collected for ppb analyses. These compounds and the spiking levels are listed below:

	PPT	PPB
Naphthalene	20 ng/L	50ug/L
Fluorene	20	50
Chrysene	20	50
Indene	20	50
Quinoline	20	50
Benzo(e)pyrene	20	50
2-methyl naphthalene	20	50

RMAL will validate the analytical data by utilizing matrix spike sample recovery criteria in conjunction with the surrogate recovery criteria. If the criteria for the matrix spike are met, only samples which do not meet the surrogate recovery criteria in that batch will be considered invalid. If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, the data will be considered valid. If the second matrix spike analysis does not meet the criteria, the data for the sample will be reported but qualified as being outside of the acceptance limits of the method. Both the original and reanalysis data will be reported.

The initial matrix spike criteria for data validity are as follows:

- o The average of the percent recoveries for all seven compounds must fall between 20 and 150 percent.
- o Only one compound can be below its required minimum percent recovery. These minimum percent recoveries are:
 - 1) 10% for chrysene, and benzo(e)pyrene, and
 - 2) 20% for all other compounds.

Criteria for data validity for each individual matrix spike compound will be developed as data is collected and will be updated on a quarterly basis.

Both matrix spike and surrogate spike recoveries will be used in assessing quality assurance/quality control for RMAL's analytical work.

11.1.5 Duplicates

The laboratory will analyze 10% duplicate samples. Percent difference between duplicates will be calculated for each detected compound.

11.2 Phenolics Analyses

11.2.1 Calibration and Analysis

The calibration and analysis procedures and acceptance criteria for the total phenolics analyses will be as described in EPA Method 420.2. A five-point calibration curve will be run daily prior to the analysis of any samples.

The calibration and analysis procedures and acceptance criteria for the acid fraction compounds in the annual GAC plant sample will be those given in the CLP protocol (SOW 7/87, Exhibits D and E).

11.3 Expanded Analyses

If expanded analyses are required in accordance with RAP Section 9.3.3, an addendum will be written to the QAPP to encompass these analyses.

12. PERFORMANCE AND SYSTEM AUDITS

RMAL participates in a wide variety of certifications, programs and contracts, and is therefore subjected to rigorous external performance evaluations and audits by the EPA, numerous other state and federal government agencies, and industrial clients. The purpose of these audits is to ensure that laboratory sample control, analysis, data and documentation meet stringent regulatory requirements and that these procedures comply with good laboratory practices.

In addition to external audits and site visits, RMAL is subject to the following Enseco (RMAL's corporate partner) audits:

1. Weekly walk-throughs by the laboratory QA Officer and Safety Officer
2. Monthly systems audits conducted by the laboratory QA Officer
3. Quarterly audits conducted by the Corporate Vice President of Quality Assurance
4. Special audits by the laboratory QA Officer and Corporate Vice President of Quality Assurance when problems are identified.

Another form of evaluation is the analysis of blind samples, a procedure important to assessing the true quality of the analytical system. As participants in the EPA Contract Laboratory program (CLP) and other contracts and certifications, RMAL is required to analyze blind samples for organics and inorganics on a quarterly basis.

In addition to mandatory blind samples from regulatory agencies, RMAL routinely analyzes internal check samples as described below:

1. Laboratory control samples (LCS) and surrogate control samples (SCS).
2. Samples originally submitted to RMAL are resubmitted as blind samples to either RMAL or to other Enseco laboratories for comparison. The results are evaluated by the Corporate Vice President of Quality Assurance, the Laboratory QA Officer, and senior staff scientists.
3. An independent commercial firm is contracted to provide all Enseco laboratories with blind check samples on a quarterly basis. Results of such samples are evaluated by both the outside firm and by Enseco's Corporate Vice President of Quality Assurance.

The following pages summarize the certification status of RMAL on a national basis. The majority of these certifications require the successful completion of performance evaluation samples and laboratory audits.

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ROCKY MOUNTAIN ANALYTICAL
a Division of ENSECO, INC.

NATIONAL CERTIFICATION STATUS

1. State of Colorado:

Agency: Colorado Department of Health

Lab ID: C0026

Effective Date: August 10, 1984

Expiration Date: renewal in January, 1987

Permit for: Monitoring Drinking Water Samples (SDWA)

Analyses:

Nitrate

Fluoride

Trace Metals (As, Ba, Cd, Cr, Pb, Hg, NO₃, Ag, Na)

Chlorinated hydrocarbons (Endrin, Lindane,
Methoxychlor, Toxaphene)

Chlorophenoxys (2,4-D; 2,4,5-TP Silvex)

Total Trihalomethanes (Bromodichloromethane,
Dibromochloromethane, Bromoform, Chloroform)

Comments: Letter on file in QA office

2. State of Florida:

Agency: Department of Health and Rehabilitative Services

Lab ID: 87278

Effective Date: July 1, 1986

Expiration Date: renewal June 30, 1987

Permit for: Monitoring Drinking Water samples (SDWA)

Analyses:

Primary Inorganic: Fluoride

Trace Metals as in (1) above

Secondary Inorganic Parameters

Organic: Chlorinated hydrocarbons as in (1) above

Chlorophenoxy acids as in (1) above

Trihalomethanes: As in (1) above

Volatile Organic Compounds

Purgeables

Pesticides

Base Neutral/Acid Extractables

Comments: Letter, certificate on file in QA office

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National Certification Status (cont.)

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3. State of Utah:

Agency: Utah Department of Health
Lab ID: Certificate No. E-83 Class I
Effective Date: June 2, 1987
Expiration Date: renewal annually pending successful completion
of WS, WP and/or Utah Department of Health PE
samples.
Permit for: Analysis of Environmental samples
Analyses:
 Trace metals
 Minerals
 Nutrients
 Demand
 Organic: Herbicides, Pesticides, PCB's, TOX, Priority
 Pollutants, Trihalomethanes, Volatile Organics
 Miscellaneous: EP-Toxicity, Solids, Sulfides, Phenols,
 Turbidity, Corrosivity, Res. Chlorine

Comments: Letter and certificate on file in QA office.

4. State of New York

Agency: Department of Health
Lab ID: 10809
Effective Date: September 23, 1986
Expiration Date: 12:01 AM April 1, 1987
Permit for: Approval for Potable and Non-Potable analyses

Comments: Ned Smith, Program Administrator, informed RMAL that this certification must be recognized by all agencies in New York. A six page list of parameters included in this certification is on file in the QA office. This certification is INTERIM pending successful analysis of PE samples twice annually.
An audit was conducted February 3-4, 1987.

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5. State of New York

Agency: Department of Environmental Conservation
Lab ID: N/A
Effective Date: N/A
Expiration Date: N/A
Permit for: N/A

Comments: IN PROGRESS. PE sample results (RMAL #61942) submitted 9/23/86. Certification granted by this agency is NOT recognized by the State Department of Health, and is only valid for contracts awarded by this agency. Laboratory audit is scheduled for January 21, 1987. Performance evaluation samples were satisfactorily analyzed. On January 22, 1987, representatives of this agency were at RMAL to discuss the possibility of extending ERCO's current contract to have RMAL absorb the overflow work. This arrangement most likely will begin March, 1987.

6. State of New Jersey:

Agency: Department of Environmental Protection
Lab ID: 45556
Effective Date: N/A
Expiration Date: N/A
Permit for: Perform Water Pollution Analyses

Comments: IN PROGRESS. RMAL is currently certified for EPA-CLP work ONLY. Performance evaluation samples (RMAL nos. 62071, 62104) currently in house were satisfactorily analyzed. Full certification pending receipt of method validation work for methods 601, 602, 608, 612, 624, 625 and an unannounced lab audit by January 31, 1987. Method validation work for 601, 602, 608, 624, and 625 complete. Awaiting results for 612.

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7. US Army Corps of Engineers:

Agency: US Army Corps of Engineers

Lab ID: N/A

Effective date: (will be processed based on RMAL performance on 61647 PE, and a lab audit)

Expiration Date: (unknown at present)

Permit for: Analysis of water and soil samples

Analyses:

RCRA Metals (Sb,As,Ba,Be,Cd,Pb,Hg,Ni,Se,Ag,Tl)

Organochlorine Pesticides, Method 608 (Aldrin, BHC, Chlordane, DDD, DDE, DDT, Dieldrin, Endosulfans, Endrin, Heptachlor, Heptachlor epoxide, Toxaphene)

PCB's

Volatiles, Method 624

Base/Neutral/Acids, Method 625

Comments: PE samples completed and results submitted 7/21 and 8/1. Richard Karn from the Corps of Engineers informed RMAL on 8/20/86 that all results for the performance samples RMAL project 61647 were within acceptance criteria.

8. US Army Toxic and Hazardous Materials Agency (USATHAMA)

Agency: USATHAMA, Aberdeen Proving Ground, MD

Lab ID: N/A

Effective Date: June 1986 (retroactive to July 1985)

Expiration Date: N/A

Permit for: Extraction and analysis of Soil samples

Analyses:

Semivolatile Organics in soils/sediments

Volatile Organics in soils/sediments

Metals in soil by ICP

Arsenic in soil by Graphite Furnace AAS

Mercury in soil by Cold Vapor AAS

DBCP in soil by GC/ECD

Fluoride in soils by ISE

Comments: USATHAMA certification is only applicable to USATHAMA projects. Once obtained, certification can be used in conjunction with any project identified by the project commander

National Certification Status (cont.)

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9. Contract Laboratory Program (CLP):

Agency: USEPA

Lab ID: N/A. . . All contracts registered under RMAL

Effective Date: RMAL has been involved since inception in 1980

Expiration: Certification is obtained on a contract basis and expires with that contract. Each contract requires a pre-award evaluation.

Permit for: RAS work for Organic Inorganic parameters and Dioxin. RMAL has performed SAS work on occassion. All environmental samples

Analyses: Work under CLP guidelines consists of:
All priority pollutants listed under Clean Water Act
HSP compounds:

Volatile Organics . . Purge/Trap--GC/MS
Semivolatile Organics . . Extraction--GC/MS
Pesticides and PCB's . . Extraction--GC/ECD
Metals (& Cyanide) . . ICP/GFAAS

Comments: RMAL is one of only five laboratories that have participated in all three areas. RMAL is frequently consulted regarding changes in methodologies, and is a well respected participant.

10. CDC Toxicology Blood Lead Proficiency Program

Agency: OSHA-Center for Disease Control (CDC)

Lab ID: 050043

Effective Date: 3rd quarter 1986

Expiration Date: This program is being deleted in 1987

Permit for: Analysis of Blood Lead Content

Analysis: Lead in Blood (GFAAS)

Comments: The most recent list of approved laboratories (March 3, 1986) is titled the September 1985 List and covers Quarters 1, 2 & 3 for 1985) this list expired March 7. In order to be an approved laboratory, a minimum of 8 out of 9 PE samples in 3 consecutive quarters must fall within acceptance limits. RMAL scored 2 of 3 correct for Quarter 4, 1985, as well as 3 of 3 for Quarter 1 & 2, 1986. This qualifies RMAL for certification, and the next list will reflect this. The CDC will confirm certification status by phone in the interim. This program will end with the first quarter, 1987.

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11. NPDES: Discharge Monitoring & Reporting QA (DMRQA)

Agency: USEPA
Lab ID: Anaconda Minerals (C00029793)
Effective Date: N/A
Expiration Date: Annual re-evaluation
Permit for: Analysis of water samples for NPDES Inorganics
Analyses:
Metals: Sb,As,Be,Cd,Cr,Cu,Pb,Hg,Ni,Se,Ag,Tl,Zn (Total)
Cyanides
Phenols
Conventional Pollutants: Br, Res. Cl, Color, Coliforms,
F,NO3,Org. N,O&G,TP,S04,S=S03,MSAB,Al,Ba,B,Co,Fe,
Mg.Mo,Mn,Sn,Ti (Total)

Comments: The results for DMR QA studies 004, 005, and 006 are on file in the QA office. RMAL analyzes PE samples annually and uses the multiple permit option to report the results to several clients. An audit was performed by the Region VIII EPA and Frontier Oil on December 9 and 15, 1986. Several minor compliance problems were discovered. These problems have since been corrected.

12. State of Oklahoma

Agency: Water Resources Board
Lab ID: 8614
Effective Date: January 1, 1987 (PENDING)
Expiration Date: June 30, 1987
Permit for: Analysis of environmental samples

Comments: A complete list of certified parameters is available in the QA office as well as a certificate from the state, and a complete report on performance evaluation results.

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13. State of Oregon:

Agency: Department of Health
Lab ID: N/A
Effective Date: N/A
Expiration Date: N/A
Permit for: N/A

Comments: IN PROGRESS. RMAL has contacted the state office, and was told that repiprocal certification may be granted if Colorado's regulatory criteria are as stringent as those in Oregon. Acceptable performance on all parameters within the USEPA Water Supply Series (WS) and Water Pollution Series (WP) is required at least once annually. An application was filed in October 1986. A follow-up telephone call in January of 1987 was made to inquire about the status. The Agency Director informed RMAL that the issue of allowing out-of-state laboratories had been passed to the Attorney General for a ruling. An off the record opinion was offered that such certifications would only be given to contiguous states.

14. State of California

Agency: Department of Health Services
Lab ID: N/A
Effective Date: N/A
Expiration Date: N/A
Permit for: Hazardous Waste Testing Certification

Comments: Tony Wong has been negotiating for all ENSECO labs to be certified by this agency. He is only awaiting their acceptance of his proposal that all labs will meet CAL's QA windows for accuracy and precision.

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15. Permit to Move Quarantined Soil

Agency: US Department of Agriculture

Permit No.: S-2899

Lab ID: N/A

Effective Date: November 15, 1985

Expiration Date: November 30, 1990

Permit for: To receive shipments of soil samples from foreign countries
for laboratory analysis. Applicable quarantines are: (80)
Witchweed, (81) Imported Fire Ant, and (85) Golden Nematode

Comments: Soil samples received under this permit must be enclosed in
polyethylene containers, and be less than 1 lb. in weight. All
samples must be kept under locked chain of custody while in
possession, and must be disposed of by incineration at a
hazardous waste facility.

13. PREVENTIVE MAINTENANCE

Since instrumental methods of analysis require properly maintained and calibrated equipment, the operation and maintenance of modern analytical instrumentation is of primary importance in the production of acceptable data. In order to provide this data, RMAL subscribes to the following programs:

- o maintenance agreements/service contracts with instrument manufacturers
- o laboratory preventive maintenance program

13.1 Service Contracts

Analytical equipment utilized by RMAL laboratory personnel for this project are covered by maintenance agreements with the instrument manufacturers. These manufacturers provide for both periodic "preventive" service calls as well as the non-routine or emergency calls.

13.2 Instrument Logbooks

Individual instrument logbooks are maintained for each piece of equipment and located near the instrument. General information contained in the logbooks include:

- o Inventory information:
equipment name, model number, serial number, manufacturer, date of acquisition, original cost
- o Service tasks and intervals:
cleaning, calibration, operation based on the manufacturer's recommended schedule, and previous laboratory experience
- o Service record:
date of breakdown, date of return to service, downtime, problems, repairs, cost of repairs, who performed the repairs, parts required, etc.
- o calibration/performance checks
- o daily operational notes

Analysts are referred to manufacturers' operating manuals for specific procedures to be followed in the operation and/or maintenance of the individual instruments.

Laboratory preventive maintenance includes any tasks that can be performed in-house, i.e., systematic cleaning of component parts as recommended in the instrument manual. If problems cannot be corrected by laboratory personnel, the instrument service representative is contacted and a service call requested to correct the problem.

14. SPECIFIC PROCEDURES TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

A quality control program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these limits to detect errors or out-of-control events, and requiring corrective action techniques to prevent or minimize the recurrence of these events.

14.1 External and Internal Components

The accuracy and precision of sample measurements are influenced by both external and internal factors. External factors or errors are those associated with field collection and sample transportation. Internal factors or errors are those associated with sample preparation and analysis. External factors are defined briefly in Section 14.1.1. Internal factors are defined in Section 14.1.2. These internal components associated with laboratory practices, procedures, and controls of data quality confidence are presented in further depth.

14.1.1 External Components: Accuracy and Precision Measurements

The results for quality control samples taken in the field represent the best estimates of accuracy and precision for the samples, since these values reflect the entire process from sample collection through sample analysis. Below is a brief description of the information provided by each of these control samples:

- o Field matrix spike - provides an estimate of bias based on recovery; includes matrix effects associated with sample preservation, shipping, preparation, and analysis.
- o Field collected samples or replicates - independent samples collected at the same point in space and time. These give the best measurement of precision for sample collection through analysis.
- o Field duplicate - a sample that has been divided into two or more portions. The analytical values obtained for each of these portions gives a second best measurement of precision for the entire sampling and analysis scheme.

14.1.2 Internal Components: Accuracy and Precision Measurements

The results of quality control samples created in the laboratory represent estimates of analysis and precision for the preparation and analysis steps of sample handling. This section describes the quality control-type information provided by each of these analytical measurements. The frequency of each of these measurements is discussed in Section 11.0, Internal Quality Control Checks.

Accuracy Measurements

- o Laboratory fortifications - provide an estimate of bias based on recovery of the compounds analyzed for the sample batch, incorporating matrix effects associated with sample preparation and analysis.
- o Surrogates - provide an estimate of bias based on recovery of similar compounds, but not the compounds analyzed, for each sample, incorporating matrix effects associated with sample preparation and analysis.
- o Internal standard - an analyte that has the same characteristics as the surrogate, but is added to each sample in a batch, just prior to analysis. It measures bias or change in instrument performance from sample to sample, incorporating matrix effects associated with the analysis process only.
- o Analysis matrix spikes - The analysis matrix spike is added prior to analysis. These spikes are similar to the internal standard; however, the analyte used is the same as that being analyzed and usually is added to a selected few samples in a batch of analyses. It incorporates matrix effects associated with the analysis step only.

Precision Measurements

- o Laboratory duplicates - a sample that has been homogenized and split into two equal portions before the method sample preparation process. It measures sample precision associated with the preparation through analysis.
- o Analysis replicate - a sample solution or extract that has been split before analysis; measures sample precision associated with the analysis only

15. CORRECTIVE ACTION

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is somewhat dependent on the analysis and the event.

Generally, out-of-control events or potential out-of-control events are noted on an out-of-control event form (see Figure 15-1). This form is part of the data package and, thus, must be completed prior to data approval. If an out-of-control event does occur during analysis, for instance, a surrogate recovery falls outside the expected range, the analyst must describe on this form: the event, the investigative and corrective action taken, and the cause of the event, and notify the Laboratory Quality Control Director. In some cases, investigation of an out-of-control event will reveal no problems. In such cases, only the event and the investigative action is recorded. If an out-of-control event is discovered during data package review, the Laboratory Quality Control Director notifies the supervisor for corrective action.

15.1 Low-level PAH Analyses/Extended Analyses/Non-Criteria PAH Analyses

15.1.1 Surrogates

The laboratory will use the surrogates: naphthalene-d₈; fluorene-d₁₀ and chrysene-d₁₂ at a sample concentration level of 10 ng/L (ppt) or 20 ug/L (ppb). RMAL will calculate the percent recovery of each surrogate for each sample. Corrective action will be taken whenever the surrogate recovery for any one or more surrogates is outside the following acceptance criteria:

<u>Surrogate</u>	<u>Acceptance Criteria %</u>	
	<u>Low-level</u>	<u>Non-criteria</u>
Naphthalene-d ₈	14-108	25-175
Fluorene-d ₁₀	41-162	25-175
Chrysene-d ₁₂	10-118	25-175

The surrogate recovery acceptance criteria will be updated quarterly.

The following corrective action will be taken when required as stated above:

- Check calculations to assure there are no errors;
- Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
- Reanalyze the sample or extract if the steps in part a) or b) fail to reveal a problem. If reanalysis of the extracts yields surrogate spike recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported.

CORRECTIVE ACTION REPORT

PROBLEM

(Briefly describe problem and QC Lot involved)

ANALYST RESPONSE

(Describe corrective actions taken and results)

Analyst:

Date:

SUPERVISOR COMMENTS

(Comment on corrective measures taken. Evaluate the effect of the problem on sample data.)

Supervisor:

Date

QUALITY ASSURANCE APPROVAL

(Comment on any necessary follow-up.)

QA Coordinator:

Date:

- d) If a), b) or c) do not correct the problem, the data for that sample will be reported but will not count towards satisfying the monitoring requirements of the RAP.

15.1.2 Matrix Spikes

RMAL will use seven representative compounds spiked into a sample of water collected in the field. These compounds and the spiking levels are listed below:

	PPT	PPB
Naphthalene	20 ng/L	50ug/L
Fluorene	20	50
Chrysene	20	50
Indene	20	50
Quinoline	20	50
Benzo(e)pyrene	20	50
2-methyl naphthalene	20	50

The initial matrix spike criteria for data validity are as follows:

- o The average of the percent recoveries for all seven compounds must fall between 20 and 150 percent.
- o Only one compound can be below its required minimum percent recovery. These minimum percent recoveries are:
 - 1) 10% for chrysene, and benzo(e)pyrene, and
 - 2) 20% for all other compounds.

Criteria for data validity for each individual matrix spike compound will be developed as data is collected and will be updated on a quarterly basis.

If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, the data will be considered valid. If the second matrix spike analysis does not meet the acceptance criteria, the data will be reported but qualified as being outside of the acceptance limits of the method. Both the original and reanalysis data will be reported. Both matrix spike and surrogate spike recoveries will be used in assessing quality assurance/quality control for RMAL's analytical work.

15.2 Expanded Analyses

If expanded analyses are required as stated in the RAP Section 9.3.3, an addendum will be written to this QAPP encompassing all methodological and quality control procedures.

15.3 Other Corrective Actions

These sections discuss corrective actions which will be taken in the event that a sample or sample extract is lost or destroyed during shipment, storage or analysis, or in performance and system audits.

15.3.1 Samples

In order to minimize the possibility of sample destruction during shipment, six 1-liter bottles will be taken for all low-level (ppt) samples. For all samples, field blanks, duplicates, and matrix spikes subsequent extraction and analysis will be conducted on four intact 1-liter bottles. All field blanks will be collected in duplicate. One field blank will be analyzed with the sample set and the duplicate will be extracted and held. In the event that the field blank is lost during analysis or invalidated, the duplicate field blank will be analyzed and reported.

If less than four liters of a sample remains after shipment and storage for analysis, the City will be notified and another sample will be collected and shipped to the laboratory for analysis. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

15.3.2 Sample Extracts

If a sample extract is broken or lost during analysis, the City will be notified and another sample will be collected and shipped to the laboratory for analysis if necessary, depending upon the data completeness requirements for the specific sample type. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

15.3.3 Quality Control Samples

If a method blank, or matrix spike is lost or broken during analysis, a replacement QC sample will be sampled and analyzed. The analysis report will clearly note that a replacement QC sample was analyzed.

If a field blank is lost or broken during shipment, storage, or analysis, no replacement will be analyzed. The analysis report for the sample batch associated with the field or shipping blank will clearly note in the discussion section why the data is unavailable. If the interpretation of the data from samples associated with the affected field blank warrant it, resampling of the entire batch may be conducted. This decision would be reached by concurrence of the EPA, MPCA and City project leaders.

15.3.4 Performance and System Audits

Each systems audit is immediately followed by a debriefing, in which the auditor discusses his findings with the laboratory representatives. The debriefing serves a two-fold purpose. First, laboratory management is afforded an early summary of findings, which allows them to begin formulating corrective strategies, and second, the auditor has a chance to test preliminary conclusions and to correct any misconceptions before drafting his report.

The systems audit report (which may or may not contain performance audit findings) is first issued in draft to the Laboratory Quality Control Director. The QC Director distributes the draft to the Laboratory Director and appropriate supervisors to solicit comments and/or rebuttals. These responses are forwarded, in writing, to the auditor. The auditor makes revisions to the draft, on the basis of these responses, at his discretion. Any points of disagreement between the QA department and the laboratory organization are resolved through discussion before the final report is issued. Written responses to the draft report are attached to the final report as an appendix.

Final audit reports are issued to project management and to corporate management. Items requiring corrective action are documented on a Corrective Action Request Form addressed to the project manager. One copy is retained by QA upon issuance. The project manager receives the original and one copy. When satisfactory progress has been achieved on each requested action, the project manager or designee enters descriptions of actions and results on the form, then retains the copy and returns the original to QA to close the loop.

Results of interlaboratory performance surveys and in-house audits, along with unresolved corrective action items are summarized in a quarterly report from the Quality Assurance Director to the Executive Vice President.

16. QUALITY ASSUARANCE REPORTS TO MANAGEMENT

Executing and administering an effective QA program in a large and complex laboratory system demands the skills of a highly qualified staff. The organizational structure of Enseco's (RMAL's corporate partner) Quality Assurance Group (Figure 16-1) provides a disciplined national management network which oversees and regulates all laboratory QA functions.

Enseco's Quality Assurance Group is headed by Kathleen Carlberg, Corporate Vice President of Quality Assurance, who reports directly to the Enseco Executive Committee and to the Chairman of the Board. As principal architect of Enseco's QA program, Ms. Carlberg has charted a rigid course to monitor and control laboratory operations. This involves the intricate process of developing QA manuals, QC protocols, training programs, Standard Operating Procedures (SOP's), uniform statistical data, interlaboratory and intralaboratory performance evaluation studies, and internal auditing programs. Ms. Carlberg is responsible for the administration and implementation of the QA program at all Enseco laboratories.

Laboratory QA activities are specifically designed to fulfill the requirements of both the individual laboratory and Enseco. Directing these activities as Divisional Director of RMAL, Dr. Mark Bollinger works closely with the laboratory Quality Assurance Officer, Robert Hanisch, who enforces and monitors the program.

Because a QA program undergoes its most stringent test at the laboratory level, Laboratory QA Officers hold a cornerstone position in the organizational structure. Enseco QA Officers are highly skilled analytical scientists, knowledgeable in all aspects of laboratory operations. Their responsibilities include diagnosing quality defects and resolving problems with the analytical system; conducting performance evaluation studies, inhouse audits, and walk-throughs; performing statistical analyses of data; auditing spike sample results; enforcing chain-of-custody procedures; assisting in the development of QA manual, SOP's and QC protocols; conducting QA training programs; and maintaining extensive records and archives of all QA/QC data.

Laboratory QA Officers report to both the laboratory president and to Dr. Wong. They also interface with one another in a peer evaluation and auditing system that encourages assistance and feedback, problem analysis, and collaboration on ways to improve laboratory performance.

In conjunction with the Laboratory QA Department, laboratory vice presidents, directors, and managers are responsible for a subset of QA activities, and work closely with supervisors to evaluate daily laboratory functions.

Ultimately, no plan can succeed without the cooperation and support of the entire working force. Enseco takes pride in its most valuable resource - the men and women whose unwavering dedication to excellence forms the building blocks of our success.

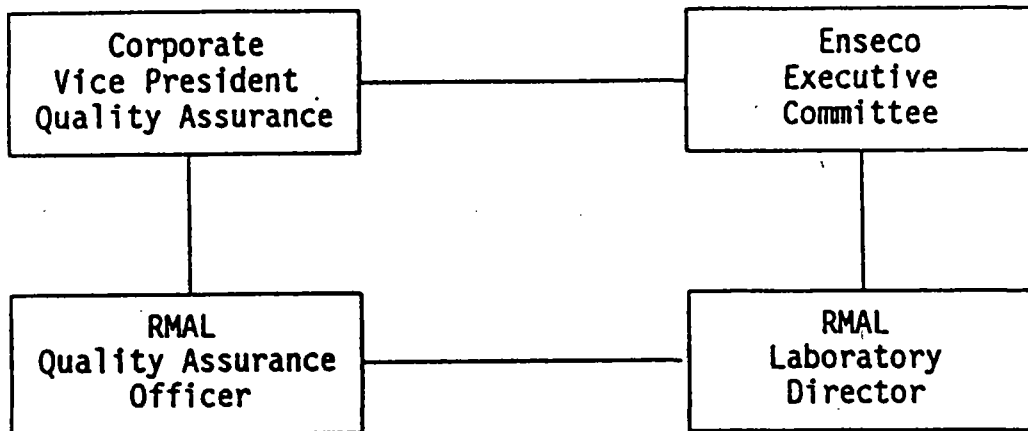


FIGURE 16-1
ENSECO QUALITY ASSURANCE GROUP ORGANIZATION CHART

A P P E N D I X A
S T A N D A R D O P E R A T I N G P R O C E D U R E S

QUALITY ASSURANCE BRANCH

APR 05 1988

ENVIRONMENT SERVICES DIVISION

INDEX OF STANDARD OPERATING PROCEDURES

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STANDARD OPERATING PROCEDURE

(REFER TO QAPP SECTION 6.7)

Title:

Calibration and Operation of Hydrolab Water Quality Monitor

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Date: 1st Qtr 1984
Number: SOP 7320
Revision: 1

1.0 Applicability

This Standard Operating Procedure (SOP) provides basic instructions to be employed for the field operation of Hydrolab digital multimeters (Model Nos. 4041 and 8000). Hydrolabs are used for field measurement of water-quality parameters.

2.0 Responsibilities

The field team is responsible for ensuring that the Hydrolab unit is in proper operating condition prior to use in the field. All system-calibration checks are the responsibility of the field team.

3.0 Materials

- Hydrolab Operation and Maintenance Instruction Manual
- Hydrolab Sonde unit, battery pack and surface unit
- Hydrolab calibration-cup
- Two Fisher-brand laboratory potassium chloride (KCl) standard solutions (known conductivity at 25°C)
- Two freshly prepared pH buffer solutions. Generally pH 7.0 and pH 4.0 or 10.0 are used.
- Distilled or de-ionized water (approximately two liters)
- Chemical-free paper towels
- Screwdrivers (as supplied in the Hydrolab Accessory Kit)

4.0 Procedures

The Hydrolab provides simultaneous measurement of four water quality parameters; 1) dissolved oxygen, in mg/l, 2) temperature, in °C; 3) pH, in standard units, and 4) conductivity, in umhos/cm (uS/cm). The panel switch on the front of the indicator unit controls which parameter is being measured and read-out.

The display is read in the following manner; temperature, pH and dissolved oxygen are read out directly. For example, a temperature of 21.8°C will be displayed as 21.8. A dissolved oxygen (D.O.) or pH reading of 8.1 will be displayed as 08.1. Conductivity is read out directly on the 2k scale. If the 20k scale is required to measure higher conductivity the number that is displayed will need to be

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multiplied by 10. In the 200k scale the reading will be multiplied by 100. For example, suppose the sample being measured has a conductivity of 1527 uS/cm. Using the 2k scale, the display will show 1527 (direct read-out). Using the 20k scale the display will show 153 ($153 \times 10 = 1530$ uS/cm). Using the 200k range the display will show 015 ($015 \times 100 = 1500$ uS/cm). Only the Hydrolab model 4041 offers the three scale measurement. The Hydrolab model 8000 is restricted to measurement of conductivity within the range of 0-2000.

4.1 Hydrolab Calibration

A complete calibration check should be performed before going to and after returning from a field sampling/water quality measurement activity. The calibration procedures should be carried out in a controlled environment such as a laboratory, but a field office or closed-in shelter may also be used.

At least one hour prior to calibration, take the following preparatory steps:

- 1) Remove the "Storage-Cup" from the Sonde Unit.
- 2) Remove the protective guard from the dissolved oxygen sensor.
- 3) Install the "Calibration-Cup" on the Sonde Unit and fill to the brim with distilled water.
- 4) Seal the Calibration Cup with the soft plastic cap and store the sonde unit, calibration standards, and the distilled water at constant room temperature for at least one hour in order to bring the various sensors, temperature compensating elements, and the calibration solutions into thermal equilibrium (within a few degrees).

All of the calibration controls are located on the front panel of the Indicator Unit. Adjustments, if necessary, should be made in the following manner:

- 1) Remove the appropriate seal-screw for the parameter being adjusted.
- 2) Insert a small screwdriver through the access hole and adjust the calibration control in the direction which brings the reading into agreement with the value of the standard solution being employed.
- 3) Replace the seal-screw.

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A RINSE STEP will be used several times during the calibration procedure. It is to be performed in the following manner: Fill the calibration cup halfway with de-ionized or distilled water. Snap on the soft plastic cap; shake the sonde unit for ten seconds and then pour out the water. Repeat twice more using fresh de-ionized or distilled water. Remove the cup and shake as much of the rinse water as possible from the electrodes.

4.1.1 Dissolved Oxygen Calibration

The Dissolved Oxygen system is the first to be calibrated since the water that has been stored in the calibration cup is used to maintain control of the temperature inside the cup. The calibration standard is either a water sample of a known D.O. concentration (determined in the laboratory by the Winkler or iodometric method in accordance with Standard Methods for the Examination of Water and Wastewater, 15th Edition, APHA-AWWA-WPCF, 1980 or water-saturated air at the temperature inside the calibration cup. The following procedures are for the water-saturated air method for D.O. calibration.

Invert the Sonde Unit and remove the soft plastic cap. Pour off enough water to bring the level to just below the D.O. membrane-retainer O-ring. With a clean paper towel or tissue blot any moisture from the D.O membrane. Cover the calibration cup mouth with one of the hard plastic caps provided in the Accessory Kit. This will keep drafts from blowing on the membrane. Do not seal the cup with the plastic cap, because that could cause a partial-pressure change in the cup. Wait approximately 5 minutes, or until the reading is stable, then switch to the TEMPERATURE position and record the temperature reading. Refer to Table 1 for the correct oxygen concentration at this temperature. Since the table values refer to concentrations at Standard Pressure it will be necessary to correct the value for local barometric pressure. This should be done in the following manner:

Correct D.O. Setting = (Local Barometric
Pressure/760mm) x (Table value
at Cup Temperature)

EXAMPLE: If T = 28.5°C and Local Barometric
Pressure = 800mm,

Correct D.O. Setting = (800mm/760mm) x (7.6 mg/l)
= 8.0 mg/l

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If a barometer is not available, the equivalent pressure may be estimated from Table 2 which relates atmospheric pressure with elevation above mean sea level. Therefore, the approximate atmospheric pressure at an altitude of 2000 feet, for example, would be: Local Atmospheric Pressure = 705mm Hg.

Adjust the Dissolved Oxygen calibration control until the proper value (rounded to nearest tenth) is displayed. Pour out the water; and then follow with a RINSE STEP.

4.1.2 pH Calibration

Calibrating the pH system requires the use of two Fisher-brand pH laboratory buffer solutions. Depending upon the application, either pH 4.0 or pH 10.0 is used in addition to pH 7.0. Invert the sonde unit and fill the calibration cup with fresh pH 7.0 buffer solution. Switch to "pH", and wait approximately 5 minutes for thermal equilibrium. Then adjust the pH calibration control until 7.0 is displayed on the read-out.

Pour out the 7.0 buffer and repeat the RINSE STEP. Invert the sonde unit and screw on the calibration cup; fill with 10.0 or 4.0 buffer. After approximately 5 minutes, adjust the pH "Slope" control until either 10.0 or 4.0 (as appropriate for the buffer being used) is displayed on the read-out. Pour out the buffer and repeat the RINSE STEP Two Times

4.1.3 Conductivity Calibration

After the second RINSE STEP, take a clean paper towel or tissue, and blot most of the moisture in the electrode area so that the standard will not suffer dilution.

Install a clean calibration cup and invert the sonde unit. The conductivity system is calibrated using at least two prepared KCl standard solutions with a known conductivity at 25°C. From Table 3, select two standard solutions with values of approximately one-third and two-thirds of the range you are most likely to encounter in the field. For example, if you are going to be working in fresh water (0-2K scale) you would want to use a 0.01M standard and a 0.005M standard. Select the more concentrated of the two standards and pour it slowly down the side of the calibration cup until full. When the reading is stable, adjust the conductivity calibration control until the display matches

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the value listed in Table 3. Empty the calibration cup and repeat the RINSE STEP Two Times. Pour in the second standard. Check the reading on the Display. It should be correct within $\pm 1\%$ of the range being used. For example, if the 0-2K scale is used, the reading for the second standard should be correct within ± 20 uS/cm of the true value. Pour out the standard solution. Perform a RINSE STEP.

4.1.4 Temperature Calibration

The temperature system is factory calibrated and is accurate to $\pm 0.2^{\circ}\text{C}$. No calibration adjustment is provided. A periodic check of the temperature system against an NBS-traceable thermometer should be performed as a verification.

4.2 Final Preparation

Turn the system off and disconnect the system components. Replace all rubber dust caps. Remove the Calibration Cup from the Sonde Unit and replace the protective guard on the dissolved oxygen electrode. Fill the Storage Cup with tap water and install onto the Sonde Unit. The system is now calibrated and ready for field use.

4.3 Field Operation

Remove the Storage Cup from the calibrated sonde unit and install the guard or the optional sample circulator. Connect the system components. Lower the sonde unit into the water (sideways, if possible) and shake it to dislodge air bubbles trapped in the conductivity cell block. Release the sonde unit and lower it to sample depth. Wait until the readings stabilize (D.O. is the best indicator) and then record the value for each parameter. Repeat at new depths or locations.

When using for ground water sampling, pour/place a sample of ground water into the Storage Cup and attach it to the sonde so that all nodes are submerged.

Check the battery voltage occasionally; charge or change batteries if the level drops below 10.5 volts. DO NOT charge the battery routinely after each day's use. Doing so may shorten the life of the battery. Use the battery until the voltage level drops to between 10.5 and 11.0 volts. At this point put the battery on charge for 24 hours.

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TABLE 1
DISSOLVED OXYGEN SATURATION VALUES IN
DISTILLED WATER AT 760 mm Hg

<u>Temp. (°C)</u>	<u>DO (mg/l)</u>	<u>Temp (°C)</u>	<u>DO (mg/l)</u>
0.0	14.6	15.5	9.9
0.5	14.4	16.0	9.8
1.0	14.2	16.5	9.7
1.5	14.0	17.0	9.6
2.0	13.9	17.5	9.5
2.5	13.7	18.0	9.4
3.0	13.5	18.5	9.3
3.5	13.3	19.0	9.2
4.0	13.1	19.5	9.1
4.5	13.0	20.0	9.0
5.0	12.8	20.5	8.9
5.5	12.6	21.0	8.8
6.0	12.5	21.5	8.8
6.5	12.3	22.0	8.7
7.0	12.1	22.5	8.6
7.5	12.0	23.0	8.5
8.0	11.8	23.5	8.4
8.5	11.7	24.0	8.3
9.0	11.6	24.5	8.2
9.5	11.4	25.0	8.2
10.0	11.3	25.5	8.1
10.5	11.1	26.0	8.0
11.0	11.0	26.5	8.0
11.5	10.9	27.0	7.9
12.0	10.8	27.5	7.8
12.5	10.6	28.0	7.7
13.0	10.5	28.5	7.6
13.5	10.4	29.0	7.6
14.0	10.3	29.5	7.5
14.5	10.2	30.0	7.4
15.0	10.0	30.5	7.4

TABLE 2

<u>Site Elevation (Feet above mean sea level)</u>	<u>Approximate Mean Barometric Pressure (mm Hg)</u>
1000	733
1500	720
2000	705
2500	694
3000	680
3500	669
4000	656
4500	644
5000	632
5500	620
6000	609
6500	598
7000	586
7500	575
8000	564
8500	554
9000	543
9500	533
10000	523

TABLE 3
CONDUCTIVITY CALIBRATION STANDARDS

Conductivities of Potassium Chloride Solutions at 25°C <u>M.W. = 74.555</u>			Conductivity Reading on Hydrolab Display for Given <u>Range Setting (uS/cm)</u>		
<u>Conc.</u>	<u>Grams KCl/L</u>	<u>uS/cm</u>	<u>(0-2K)</u>	<u>(0-20K)</u>	<u>(0-200K)</u>
0.0005	0.03728	73.9		-	-
0.001	0.07456	147.0	147	-	-
0.002	0.1491	292.0	292	-	-
0.005	0.3728	717.8	718	-	-
0.01	0.7456	1.413K	1413	141	-
0.02	1.491	2.767K	---	277	-
0.05	3.728	6.668K	---	667	-
0.1	7.456	12.90K	---	1290	129
0.2	14.911	24.82K	---	---	248
0.5	37.278	58.64K	---	---	586
1.0	74.555	111.9K	---	---	1119

NOTES:

(1) Two conductivity standards are recommended for each range setting (boxed-in values). Calibration adjustments will be made first with the higher concentration and then with the lower concentration.

(2) Single dashes indicate ranges which are not recommended for calibration checks.

(3) The Hydrolab model 8000 is restricted to conductivity readings between 0-2000 uS/cm (0-2k) scale), therefore conductivity readings and thus calibration solutions within the 0-20k and 0-200k ranges will not apply.

STANDARD
OPERATING
PROCEDURE

Subject or Title:
BUILDING SECURITY - REFER TO QAPP SECTION 7

Page 1 of 2

SOP No.:
LP-RMA-0001

Revision No.:
Original

Effective Date:
12/9/87

Supersedes:

1. Purpose:

The purpose of building security is to guarantee data security and confidentiality for the client as well as providing analytical data which is legally defensible.

2. Policies:

RMAL's security policy includes controlled access to the building, testing areas and data files, confidentially agreements with all personnel, identification badges for all personnel, electronic security and fire alarm systems, and a security guard. All visitors are also assigned visitor badges and are accompanied by RMAL staff during their stay in the facility.

3. Safety Issues: Not Applicable

4. Procedure:

Building Security

- a. All exterior doors to the facility will remain locked at all times with the exception of the front entrance.
- b. During the hours of 7:00 a.m. to 6:00 p.m., the front entrance or main reception area is controlled by the receptionist and secured by locked entries. The alarm system is not activated during this time period.
- c. During the hours of 6:00 p.m. to 7:00 a.m., the front entrance is controlled by security guard. All persons entering or leaving the facility will be recorded by the security guard. The alarm system is activated during this time period to prevent all other exterior doors from being usable, including sample receiving and the patio doors.

Prepared by:

Date:

Management Approval:

Date:

QA Officer Approval:

Date:

Robert C. Hamisch

12/9/87

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OPERATING
PROCEDUREPage 2 of 2SOP No.:
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- d. Sample receiving during the hours of 6:00 p.m. to 7:00 a.m. is permitted only with the assistance of the security guard.

Personnel Identification

- a. All employees and visitors are required to wear security badges at all times while on the premises of all ENSECO divisions.
- b. The personnel administrator is responsible for issuing a picture I.D. badge to an employee on the employee's first day of employment. Each employee is responsible for his/her badge. Additionally, each employee will be required to sign a "Confidentiality Agreement" which is included in the employee's personnel file.
- c. The receptionist is responsible for issuing a badge to each visitor to the facility. Visitors must request a badge from the front office of the division they visit, sign the visitor log and must be accompanied by an ENSECO employee before access to any building will be allowed.

Building Alarm System

- a. While it is not anticipated that employees will have to set or disarm the alarm system, it is important that employees understand the procedure. Unless used correctly, the alarm will go off and the Arvada Police Department will be called.
- b. The procedure is confidential information and can be obtained from the Personnel Department.

5. Responsibilities:

- a. It is the responsibility of each employee to maintain confidentiality of all clients data.
- b. The Personnel Department is responsible for issuing employee identification badges and having signed "Confidentiality Agreements" in each employee's personnel file.
- c. The receptionist is responsible for issuing visitor badges and for visitor sign-in during normal business hours. The security guard is likewise responsible for visitor and employee comings and goings between the hours of 6:00 p.m. and 7:00 a.m.
- d. Employees escorting visitors are responsible for ensuring that visitation procedures are followed and that data confidentiality has not been compromised.

6. Comments:

STANDARD
OPERATING
PROCEDURE

Subject or Title: Laboratory Data Review - REFER TO QAPP SECTION 10.1		Page <u>1</u> of <u>12</u>
SOP No.: LP-RMA-0002	Revision No.: Original	Effective Date: 12/9/87
Supersedes:		

1. Purpose

All laboratory data will be subjected to a rigorous data review process prior to its release to the client. The review process has been developed to minimize errors associated with sample processing, sample analysis and data reporting and to ensure that information pertaining to a given sample is well-documented. The process consists of a three-level review whereby results generated for a specific project are evaluated to ensure that

- o project is complete;
- o precision, accuracy and detection limits are met;
- o raw data interpretation is correct;
- o all calculations are correct;
- o contractual requirements are met; and,
- o all information is well documented for archival purposes.

Enseco/RMAL uses a computerized Laboratory Information Management System (LIMS), as well as a variety of custom software programs designed to perform calculations, check results, generate reports, and to ensure data integrity and security. Whenever possible, historical client-specific data may aid in the review process as an additional check on generated results.

2. Policies

All project data will be subjected to a three-tier review process including review by operations, the data review group for inorganics, GC/MS, and chromatography and the final review by the project or client managers. Data will not be released to the client until the review process is completed.

Prepared by:
Allen J. Medine, Ph.D.

Date:
December 9, 1987

Management Approval:

Date:
12/10/87

QA Officer Approval:

Date:
12/10/87

SOP No.:
LP-RMA-0002Revision No.:
OriginalEffective Date:
12/9/87

3. Safety Issues

There are no direct safety issues which are of concern for the data review process. As with other non-analytical activities, caution should always be exercised when performing data review functions in the laboratory. For example, discussing problems with analysts, examining original samples, checking preparation aliquots will require review personnel to be in the laboratory or in appropriate storage areas. A review of safety concerns for all of these areas shall be implemented.

4. Procedure

The data review framework is essentially the same for the metals, non-metals, GC/MS and chromatography groups. The differences between each groups procedure are due to analysis differences, data entry and data correction software developed for LIMS. The data review process consists of three levels (LEVEL 1, LEVEL 2 and LEVEL 3). The general framework for the laboratory review process is shown in Figure 1.

A. LEVEL 1 REVIEW

The LEVEL 1 REVIEW begins at the analytical (bench) stage where LEVEL 1 review is primarily a self-review of all information generated during the analysis. During the analytical test, the analysts have much information concerning the precision, accuracy and problems. The intent of the data review program is to take advantage of this condition by review of all analytical details generated by the analyst and subsequent approval of the test results and QC by the analysts immediate supervisor. Specifically, the functions of the analyst and supervisor are as follows:

ANALYST:

1. Review Prep Lab Notes - Preparation lab notes are to be reviewed to determine if there were anomalies observed which may affect the analysis for certain parameters.

2. Review Special Instructions - For certain projects, the Client may have specified certain modifications to a standard test, analysis using a custom test, project specific QC, or special preparation of the sample.

3. Record All Necessary Information - While this may be considered more of an operations or analytical method concern, proper documentation of the analysis, in sufficient detail to allow re-creation of the analysis, is essential for an effective, efficient data review program and to permit development of a sound data archive program. An important part of data recording is to reveal whether

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the test proceeded according to the Analytical Method SOP and that deviations from the method, anomalies during analysis, or that decisions concerning re-analysis are well-documented.

4. Check All Calculations - Errors frequently occur during calculations for standard curves, dilution factors, unit conversions, or extrapolations from instrument response to appropriate concentrations. The analysts will check ALL calculations, or verify data entry into software designed to perform calculations, examine results for agreement with expected results (i.e. order of magnitude or better) and indicate that calculations were reviewed on the LEVEL 1 REVIEW CHECKLIST.

5. Provide Data and QC Summary - Summaries of parameter concentrations and QC data generated are to be provided to the supervisor along with raw data (bench sheets, chromatograms, etc.) for supervisor approval of the analytical results.

6. Provide Out of Control/Anomaly Sheet - Information regarding out of control situations or anomalies is necessary for review personnel to re-create the analysis when there are questions concerning the data which has been generated during the analysis. Holding time violations are to be clearly indicated along with the appropriate reasons for the violation.

7. LEVEL 1 REVIEW CHECKLIST - The function of the checklist is to indicate that the above items have been considered in the analysis. The LEVEL 1 REVIEW CHECKLIST is shown in Figure 2. There are more detailed items which are considered during the analysis and the review procedure by both analysts and the immediate supervisor in the GC/MS, GC, Metals and Inorganic Groups. Much of this information can be found on the LEVEL 2 CHECKLIST's. For example, in metals analysis using graphite furnace analysis, the analysts and supervisor will examine instrument standardization criteria (absorbance for standards, etc.), dilution factors, linear range compliance, detection limit adjustment and whether the Method of Standard Additions was required.

SUPERVISOR:

It is recognized that the analyst supervisors are not a part of the data review group. However, the supervisors are directly responsible for the analytical performance of the various analyst and, as such, are an integral part of the review process. The main functions of the supervisors are to review analysis as soon as possible and 1) accept analysis or 2) suggest re-analysis. As part of

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the LEVEL 1 REVIEW process, supervisors will perform the following tasks:

1. Review analysis package for QC, reasonable results, holding tie violations and general acceptance of analysis. It is very important that re-analysis decisions be made at this level.

2. Signify approval on LEVEL 1 REVIEW CHECKLIST

3. Approval of data entry into data base management system

4. Schedule data entry (applicable to inorganics analysis at this time only).

It will be the responsibility of the supervisor to review and approve (or disapprove) the analysis on a daily basis. It will not be acceptable for supervisors to allow their review packages to stack up while other tasks are being performed. The review process depends on a continual flow of information through the the various levels. To meet turnaround times and other constraints of a commercial laboratory, it is essential for supervisors to provide a timely review of data generated by the analysts.

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12/9/87**B. LEVEL 2 REVIEW: DATA REVIEW GROUPS**

At the present time, separate data review groups exist in the inorganics division, the GC/MS division and the Chromatography division. A thorough review of the project data base takes place within the data review groups. There are numerous items which are common to each divisions review procedure. Each review group has developed a separate checklist to aid each reviewer in specifics related to the analytical tests. In addition, the reviewers in each group possess sufficient experience with the analyses conducted by the division to allow a comprehensive assessment of the precision and accuracy of the data generated.

The LEVEL 2 REVIEW is considered to be a peer review of the analytical data and review of project specific requirements. At this stage of the review, a complete check of the tests assigned to a project is made against the project data base to assess project completion. Additionally, the preparation lab notes, bench sheets, QC forms and anomaly sheets are reviewed in detail to ensure that raw data has been interpreted correctly, that detection, precision and accuracy criteria are met, that the information is well documented for archival purposes, and that contractual requirements are also met.

Each data review group will evaluate the project data with respect to the LEVEL 2 REVIEW checklists. If any re-analysis is required at this stage, the decision is documented along with other project specific data. The LEVEL 2 REVIEW CHECKLISTS for each group are shown in Figures 3-5. The completion of the LEVEL 2 REVIEW is indicated on the checklists by the appropriate signature.

The reviewers will also provide information which is used by the report preparation personnel to prepare the final project report. Reviewers should provide comments on unusual or inconsistent results, anomalies, subcontractor data, and the extent of any necessary data qualification. Reviewers are to also assemble the complete package for report generation, including the above comments and raw data, when requested.

Following the completion of the review by the peer reviewers, the complete package will be examined by the data review supervisor. Supervisors will provide additional review of comments, anomalies, data qualification, and relationships between parameters, when appropriate. Approval of the LEVEL 2 REVIEW by the supervisor is also indicated on the LEVEL 2 REVIEW CHECKLIST.

The supervisors will also check the file for completeness, address comments from reviewers, and spot check results for reasonableness. The supervisors will also develop revisions to the data review SOP, provide training to data reviewers, assist development of computer knowledge-based review software and provide a continued evaluation of data review procedures.

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At the completion of the review process for each division, the supervisor will change the project completion status in LIMS from status 4 to 7. Altering the project status in this way allows management to effectively move projects through the laboratory as rapidly as possible.

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12/9/87**C: LEVEL 3 REVIEW: CLIENT MANAGERS**

The last review of project data takes place at the client manager level. This review is directed at the results obtained, the clients needs, overall project results across analytical divisions, special instructions, analysis problems, the extent of data qualification. Client managers are not responsible for numerical errors, wrong analysis dates and other information which is the responsibility of LEVEL 1 and LEVEL 2 REVIEW.

5. Responsibilities**LEVEL 1 REVIEW**

The operations supervisors are directly responsible for the approval of the analysis and the LEVEL 1 REVIEW CHECKLIST. The analysts are responsible for the analyst items on the checklist and being aware of what takes place during LEVEL 2 REVIEW.

LEVEL 2 REVIEW

The peer reviewers in each data review group (inorg., GC/MS and chromatography) are responsible for the detailed review of all project information as indicated on the LEVEL 2 REVIEW CHECKLIST. The data review supervisor is responsible for a brief examination of the project data and comments, additional comments appropriate for the final report, training reviewers, and developing review procedures to be used for the LEVEL 1 and LEVEL 2 REVIEW.

LEVEL 3 REVIEW

The client managers are responsible for ensuring that the client's needs have been met, that the data appears reasonable and that contractual requirements have been met.

6. Comments

For the review process to be effective in correcting problems and improving data generated in the laboratory, it is essential that reviewers inform operations supervisors and client managers on a regular basis of the problems which have been identified during the review process. Review checklists or written memos would be an effective means for alerting various personnel on problems which could be avoided or should be corrected.

LABORATORY DATA REVIEW FRAMEWORK

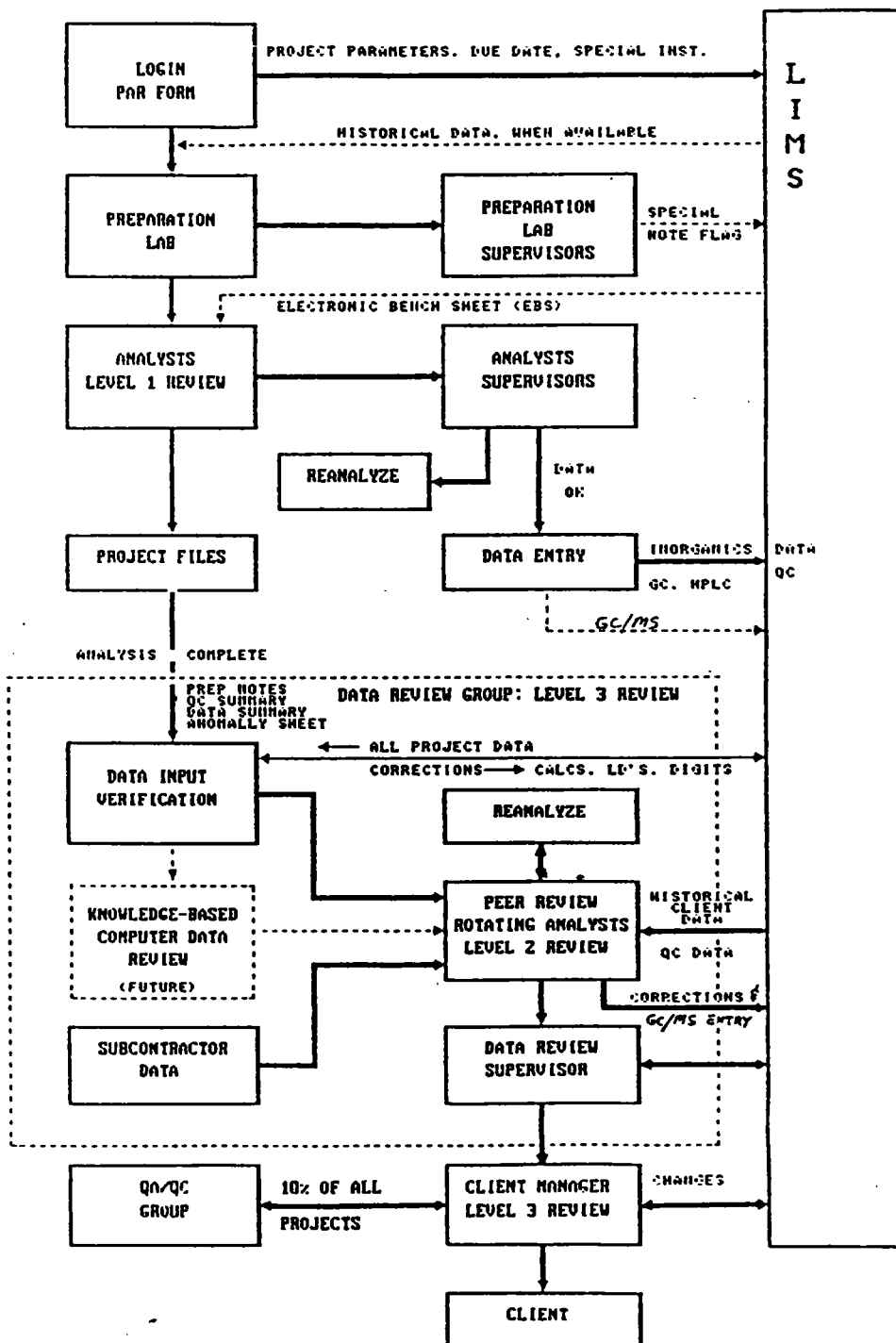


Figure 1 - Laboratory Data Review Framework Form

DATA REVIEW PROGRAM

LEVEL 1 REVIEW CHECKLIST

PROJECT # _____

ANALYTICAL TEST _____

ANALYST ITEMS

Y	N	NA	Preparation Lab Notes Reviewed
Y	N	NA	Special Instructions Followed
Y	N	NA	Samples Properly Preserved and in Proper Container
Y	N	NA	Bench Sheets (Data Package Completed With All Information, Including Special Instructions
Y	N	NA	Blank Correction Procedure Followed
Y	N	NA	All Calculations Checked
Y	N	NA	QC Within Limits
Y	N	NA	Out of Control Form Filed
Y	N	NA	Analysis Anomalies Noted

ANALYST COMMENTS:

ANALYSTS REVIEW _____ DATE _____

SUPERVISOR ITEMS

Y	N	NA	Results Appear Reasonable
Y	N	NA	Re-run Decision Documented
Y	N	NA	Holding Time Violations Documented

SUPERVISOR COMMENTS:

SUPERVISOR APPROVAL _____ DATE _____

DATE DATA ENTERED _____ BY WHOM _____

Figure 2 - Data Review Program Form

DATA REVIEW PROGRAM

LEVEL 2 REVIEW CHECKLIST

PROJECT # _____

INORGANICS: METALS _____ NON-METALS _____

Y N NA Project Assignment Record (LIMS) vs. Actual Data

Y N NA Preparation Lab Notes Reviewed

Y N NA Special Instructions Followed, Check Item

_____ Project Specific QC
_____ Raw Data Requested
_____ Limited Sample Volume
_____ Special Preparation Needed
_____ Custom Analytical Test
_____ Special Holding Times
_____ Other _____

Y N NA Bench Sheets (Analysis Package) Complete

Y N NA Special Instructions Noted

Y N NA Detection Limits Correct

Y N NA Blank Correction Procedure Followed

Y N NA Significant Digits Correct

Y N NA All Calculations Checked

Y N NA QC Checked and Acceptable

Y N NA QC Lot Assignment Correct

Y N NA Out of Control Form Filed

Y N NA Analysis Anomallies Noted

Y N NA Re-run Decision Documented

Y N NA Analysis Date Reflects Date of Accepted Data

Y N NA Holding Time Violations Documented

Y N NA Camera-Ready Report Cover Sheets Completed

Y N NA Prep sheet Attached

Y N NA Analysis Anomaly Sheet Attached

Y N NA Raw Data Attached

LEVEL 2 REVIEW APPROVAL _____ DATE _____

CORRECTIONS ENTERED _____ DATE _____

SUPERVISOR APPROVAL _____ DATE _____

GC/MS DATA REVIEW CHECKLIST

- ☐ 1. Check LIMS Test vs SOP.
- ☐ 2. Check anomalies sheet and QC forms.
- ☐ 3. Check standard and see if it was updated.
- ☐ 4. Look at chromatogram for:
 - ☐ a. carry-over
 - ☐ b. truncating peaks
 - ☐ c. general chromatographic quality
 - ☐ d. very large unknown peaks
- ☐ 5. Recalculate run factors.
- ☐ 6. Check surrogates.
- ☐ 7. Check Quant list for:
 - ☐ a. linear ranges
 - ☐ b. co-eluting compounds
 - ☐ c. IS areas
 - ☐ d. carry-over
- ☐ 8. Check spectra for ID's and saturation.
- ☐ 9. Check if TID's are pulled if necessary.
- ☐ 10. Check chromatogram vs Quant list vs TID's.
- ☐ 11. Recalculate all target compounds and TID's.
- ☐ 12. Note any anomalies not on form already.
- ☐ 13. Over-all project review (compound types, ratios).

DATA REVIEW PROGRAM

LEVEL 2 REVIEW CHECKLIST

PROJECT # _____

CHROMATOGRAPHY

Y N NA Project Assignment Record (LIMS) vs. Actual Data

Y N NA Preparation Lab Notes Reviewed

Y N NA Special Instructions Followed, Check Item

_____ Project Specific QC

_____ Raw Data Requested

_____ Limited Sample Volume

_____ Special Preparation Needed

_____ Custom Analytical Test

_____ Special Holding Times

_____ Other _____

Y N NA Bench Sheets (Analysis Package) Complete

Y N NA Special Instructions Noted

Y N NA Detection Limits Correct

Y N NA Blank Correction Procedure Followed

Y N NA Significant Digits Correct

Y N NA All Calculations Checked

Y N NA QC Checked and Acceptable

Y N NA QC Lot Assignment Correct

Y N NA Out of Control Form Filed

Y N NA Analysis Anomalies Noted

Y N NA Re-run Decision Documented

Y N NA Analysis Date Reflects Date of Accepted Data

Y N NA Holding Time Violations Documented

Y N NA Camera-Ready Report Cover Sheets Completed

Y N NA Prep sheet Attached

Y N NA Analysis Anomaly Sheet Attached

Y N NA Raw Data Attached

LEVEL 2 REVIEW APPROVAL _____ DATE _____

CORRECTIONS ENTERED _____ DATE _____

SUPERVISOR APPROVAL _____ DATE _____

STANDARD
OPERATING
PROCEDURE

Subject or Title:
SAMPLE LOG-IN - REFER TO QAPP SECTION 7.1

Page 1 of 8

SOP No.:
LP-RMA-0003

Revision No.:
Original

Effective Date:
12/9/87

Supersedes:

1. Purpose:

To create analyses records in the laboratory computer for notification to lab analysts.

2. Policies:

Log-in must be completed within 24 hours of authorization by a project manager. Authorization occurs when a PAR, Menu of Analytical Services (Figure 1), and Project Screen in the computer are filled out and the information is turned in to the Receiving area (Client Services).

3. Safety: Not Applicable

4. Procedure:

As log-in proceeds fill out the LOG-IN checklist (Figure 2) and address all the issues on this form.

- a. Retrieve the samples from the walk in cooler. Compare the bottles with the paperwork and PAR. Check for the correct test and sample matrix and properly preserved bottles for tests.
- b. Check that the labeling was done correctly and referenced properly on the chain of custody.
- c. Read the Menu of Analytical Services and PAR. Check for any jobcodes and special instructions. Special instructions are entered in the computer using a text editor and are then visible to all analysts. These are entered by the project managers.
- d. Go to Data Set Maintenance in the Computer. Modify the project screen to change the project from P(planned) to A(active). Verify the number of samples and the amount of \$.
- e. Using the group code editor in LIMS (laboratory computer) set up groups of tests according to the PAR. Duplicate and Matrix spiked samples (Project specific QC) need to have separate groups of tests. Print copies of these group codes.

Prepared by:

Robert C. Hamisch

Date:

12/10/87

Management Approval:

Chad Weber

Date:

12/10/87

QA Officer Approval:

Robert C. Hamisch

Date:

12/9/87

SOP No.:
LP--RMA-0003Revision No.:
OriginalEffective Date:
12/9/87

- f. Go to the Log-in sample program. Assign the proper group code (groups of tests) to each sample separately, adding the received date, collections date, and client identification. List the bottles received for each sample and the location they will be placed. Samples placed on a hold status may or may not be logged in depending on the decision by the project manager.
- g. Print a list of the samples, tests assigned, and bottles received through a select report. Print a copy of the project screen. Print an Acknowledgement letter indicating the samples received, RMA numbers, and any discrepancies noted upon receipt.
- h. Perform any compositing, filtering, or splitting necessary. Create any additional preserved bottles if necessary.
- i. Fill out the Analysis request form for subcontract work (Figure 3) if necessary. A purchase order must also be filled out. For subcontracting to another Enseco facility fill out the Interlaboratory Analysis Request form (Figure 4).
- j. Put samples in the proper locations. Volatiles are placed in refrigerators near the MS and GC areas. Waters for organic prep are placed in refrigerators near the Organic prep labs. Inorganic water bottles are placed in the walk in cooler and arranged by type of preservative. Solids and Wastes are stored in the walk in refrigerator on color coded shelves that are cross referenced by a color coded board in the receiving area.
- k. All printed paperwork is placed in the project folder and it is reviewed by the supervisor. The folder is then stored in the Receiving area while the project is active.
- l. Changes that need to be made to a project after log-in must be requested by filling out the Log in Change form (Figure 5).

SOP No.:
LP-RMA-0003

Revision No.:
Original

Effective Date:
12/9/87

5. Responsibilities:

Project manager is responsible for correctly filling out the PAR, special instructions, Menu of Analytical Services, and Project screen. Turnaround time is decided by the project manager. Sample Receiving technicians are responsible for transferring the information to the laboratory computer.

6. Definitions:

Special instructions - Typed instructions in LIMS to the operations groups and analysts that are necessary to complete the work and can not be indicated by using one of the computer tests.

LOG IN CHECKLIST

PROJECT #: _____ ADD ON #: _____

LOGGED BY: _____

PAR COMPLETE OR UPDATED?: _____

LIST PAR PROBLEMS: _____

PROJECT SCREEN CORRECT? (# of samples, \$ amount) _____

SAMPLE MATRIX CORRECT? _____

TEST MATRIX CORRECT? _____

SHORT HOLDING TIMES ON BOARD? _____

VOAS ON BOARD? _____

CHAIN OF CUSTODY SIGNED? _____

SAMPLE BOTTLES LABELED CORRECTLY? _____

APPROPRIATE BOTTLES FOR PARAMETERS? _____

LIST BOTTLE PROBLEMS _____

VOA VIAL FOR VOA ANALYSIS? _____

SUBCONTRACT FORM FILLED OUT? _____

BOTTLES MADE TO SEND OUT? _____

BOTTLES BOXED AND PUT AWAY? _____

SPECIAL INSTRUCTIONS IN LIMS? _____

BNA OR VOA TESTS MODIFIED? _____

GROUP CODES SENT TO JEFF LOWRY? _____

Figure 2 - Log In Checklist

ANALYSIS REQUEST FOR SUBCONTRACTS

RMAL Project Number: _____

Laboratory to be subcontracted: _____

Project Manager: _____

Results Due: _____

Sample ID Number	Matrix	Parameter	Bottle Description	Comments
------------------	--------	-----------	-----------------------	----------

Figure 3 - Analysis Request for Subcontracts

Figure 4 - Interlaboratory Analysis

LOG-IN CHANGES

DATE _____

PROJECT # _____

CHANGE REQUESTED BY _____

DESCRIBE CHANGE (tests added or deleted, corrections, etc.)

PRICE LIST INCREASE: _____

NEW DATA DUE: _____

NEW REPORT DUE: _____

PROJECT MANGER SIGNATURE _____

Figure 5 - Log In Changes

STANDARD
OPERATING
PROCEDURE

Subject or Title: USE OF PAR (Project Assignment Record) - Refer to QAPP Section 7.1 Page 1 of 29

SOP No.:
LP-RMA-0004

Revision No.:
Original

Effective Date:
12/9/87

Supersedes:

1. Purpose:

To designate and authorize the tests required for each sample (or sample site) and the matrix of these samples in order for a sample receipt technician to assign these tests in the lab computer.

2. Policies:

PAR's are always filled out before the log-in process may take place. Changing a standard list of analytes for a test logged in must be approved by a senior level manager.

3. Procedure:

- a. Choose one of the 4 types of PARs.

Long Form - for projects involving Mass spec., Chromatography, Inorganic and metal work. (Figure 1).

Inorganic - for only inorganic and metals work. (Figure 2)

Chromatography - for only chromatography work. (Figure 3)

Mass Spectrometry - for only Mass spec. work. (Figure 4)

- b. Fill in the information at top. Group the samples by similar tests required for the same sample matrix. Indicate the proper sample matrix (Figure 5). Indicate the proper test matrix (see the choices on the PAR).

For tests 01 - water
 20 - solid
 40 - waste
 16 - TCLP
 13 - EP TOX

Prepared by:

Beth Kelly

Date:

12/10/87

Management Approval:

Chad Wiley

Date:

12/10/87

QA Officer Approval:

Robert C. Harisch

Date:

12/9/87

SOP No.:
LP-RMA-0004

Revision No.:
Original

Effective Date:
12/9/87

- c. Mark the columns associated with each group of samples for the test desired (with an x). Some exceptions are:

S or Q are required for some tests to indicate single or quad analyses

T or D are required to indicate Total or Dissolved

C is required to indicate a change to a standard list. For any C marked there must be an explanation written on the PAR. For example some analytes might be deleted or added from a standard Priority Pollutant Semivolatile list.

- d. New tests that are not on the PAR must be created by the Data Administrator following completion of the Request form (Figure 6). Generic tests are available as place holders while the test is being created.

5. Responsibilities:

Project managers are responsible for the accuracy of the PAR.

6. Comments:

TCLP preps must be assigned. They are not pulled with the job codes. Some tests are not to be changed or modified (ICPLIT). Not all created tests are on the PAR. Most of the tests that RMAL sends to subcontractors must be hand written on the PAR. (Figure 7)

7. Definitions:

Jobcode - groups of tests that will be automatically assigned by the computer by the use of a simple phrase; example RCRA01C assigns all RCRA tests.

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Date: 12/9/87

Revision: Original

LONG FORM pg 1. Last Revision: 6/8/87 Current Revision: 9/15/87

Job Code Y N

Project # Proj Mgr: Prepared By: Date: / / Spcl Inst: Y N

GROUP	Smpl Mtx	Test Mtx	Client Description	RMA Sample Numbers
A				
B				
C				
D				
E				

Hazard Label:

<<< >>>	<<<A >>>	ORGANIC CHEMISTRY	<<< >>>	<<< >>>
---------	----------	-------------------	---------	---------

GC/LC Analyses	Test ID	Matrices	A	B	C	D	E
SDWA Trihalomethanes	THM SDW	01					
* Halogenated Volatile Organics **	601LI	01,20 ,16,46					
Halogenated VOA's (LOW DETECTION LIMIT)	601LL	20					
* Aromatic Volatile Organics **	602LI	01,20 ,16,46					
Aromatic VOA's (LOW DETECTION LIMITS)	602LL	20					
Benzene, Toluene, Ethylbenzene, Xylenes	602BTEX	01,20 ,16,46					
SDWA Volatiles	MCL SDW	01					
Acrolein & Acrylonitrile	603LI	01,20					
Phenols	604LI	01,20					
Benzidines	605LI	01,20					
Phthalate Esters	606LI	01,20					
Nitrosamines	607LI	01,20					
Organochlorine Pesticides/PCB's 608	OCP PP	01,20 ,16,46					
OC Pest's/PCB's (LOW DETECTION LIMIT)	OCP PPL	20					
NPDES Organochlorine Pesticides/PCB's	OCP PP	01,20					
* HSL Organochlorine Pesticides/PCB's **	OCP HSL	01,20					
HSL OCP's/PCB's (LOW DETECTION LIMIT)	OCPHSL	20					
CLP/HSL Organochlorine Pesticides/PCB's	OCP CLP	01,20					
Appendix 8 or 9 Organochlorine Pest/PCB's	OCP AP9	01,20					
TCLP Characteristic Organochlorine Pests.	OCP TCLP	01,20,16,46					
* SDWA Organochlorine Pesticides	OCP SDW	01					
* RCRA Organochlorine Pesticides	OCP RCR	01,20					
PCB's	PCB	01,20, 45					
Nitro-Aromatics & Cyclic Ketones	609LI	01,20					
* Polynuclear Aromatic Hydrocarbons / 610	LC PNA	01,20 ,16,46					
Haloethers	611LI	01,20					
Chlorinated Hydrocarbons	612LI	01,20					
Organophosphate Pesticides **	OPP	01,20					

** Most Modifiable Test

C - Change Noted

* Preferred Standard Product (01 & 20 Bolded)

01 & 20 Bolded - Std.Prd. DL

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 2.

Last Revision: 6/8/87 Current Revision: 9/15/87

GC/LC Analyses (cont.)	Test ID	Matrices	A	B	C	D	E
Appendix 8 or 9 Organophosphate Pesticides	OPP AP9	01,20					
Appendix 8 or 9 Herbicides	HRB AP9	01,20					
TCLP Herbicides	HRBTCLP	01,20, 16,46					
* SDWA Herbicides	HRB SDW	01					
* RCRA Herbicides **	HRB RCR	01,20					
Triazines	619LI	01,20					
Carbamate & Urea Pesticides/ HPLC	632LI	01,20					
Penta & Tetrachlorophenol	PCP	01,20					
Ethylene Dibromide (EDB)	504LI	01,20					
Hydrocarbon Scan by FID	GC HYD	01,20					
Boiling Point Distribution By GC	GC BPD	01,20					
Water Miscible Solvents	GC DAI	01					
Semivolatiles by MSD	MSD BNA	01,20					
Volatiles by MSD	MSD VOA	01,20					
Semivolatiles by FID	GC BNA	01,20					
Base Neutrals by FID	GC BN	01,20					
Acids by FID	GC ACD	01,20					
Land Treatment Demonstration / HPLC	LC LUD	01,20,43, 16,46					
GC / MS Analyses							
Priority Pollutant Volatiles	VOA 624	01,20 ,40 16,46					
Pri. Pollutant VOA's (LOW DETECTION LIMIT)	VOA624L	20					
Priority Pollutant Semivolatiles	BNA 625	01,20 ,40 16,46					
Priority Pollutant Acid Organics	ACD 625	01,20 ,40					
Priority Pollutant Base/Neutral Organics	BN 625	01,20 ,40					
* Hazardous Substance List Volatiles	VOA HSL	01,20 ,40 16,46					
HSL Volatiles (LOW DETECTION LIMIT)	VOAHSL	20					
* Hazardous Substance List Semivolatiles	BNA HSL	01,20,40 16,46					
* Hazardous Substance List Acid Organics	ACD HSL	01,20 ,40					
* Hazardous Substance List Base/Neutral Org	BN HSL	01,20,40					
CLP/HSL Volatiles (TID's Included)	VOA CLP	01,20,25					

** Most Modifiable Test

C-Change noted.

Bolded 01 & 20 - Std.Prd.

* Preferred Standard Product (01 & 20 Bolded)

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 3.

Last Revision: 6/8/87 Current Revision: 9/15/87

GC / MS Analyses (cont.)	Test ID	Matrices	A	B	C	D	E
CLP/HSL Semivolatiles (TID's Included)	ENA CLP	01,20,25					
NPDES Volatiles Organics	VOA 624	01					
NPDES Semivolatiles Organics	ENA 625	01					
Appendix 8 or 9 Volatiles	VOA AP9	01,20,40, 16,46					
Appendix 8 or 9 Semivolatiles	ENA AP9	01,20,40, 16,46					
Appendix 8 or 9 Chlorinated Dioxins & Furans	DXN AP9	01,20 Div.22					
CLA-CLB Dioxins & Furans	DXN	01,20 Div.22					
Appendix 8 TID Volatiles	VOATID8	01,20,40, 16,46					
Appendix 8 TID Semivolatiles	ENATID8	01,20,40, 16,46					
TCLP / Waste Characteristic Volatiles	VOATCLP	01,20,40, 16,46					
TCLP / Waste Characteristic Semivolatiles	ENATCLP	01,20,40, 16,46					
TCLP / Land Restriction Volatiles	VOA LFR	01,20,40, 16,46					
TCLP / Land Restriction Semivolatiles	ENA LFR	01,20,40, 16,46					
Refinery Hazardous Constituents Volatiles	VOA REF	01,20,40, 16,46					
Refinery VOA's (LOW DETECTION LIMIT)	VOAREFL	20					
Refinery Hazardous Constituents ENA	ENA REF	01,20,40, 16,46					
Polynuclear Aromatic Hydrocarbons	EN PNA	01,20,40, 16,46					
Polynuclear Aromatic Hydrocarbons SIM	ENSIPNA	01,20,40, 16,46					
Tentative Identification Volatiles	VOA TID	01,20,40, 16,46					
Tentative Identification Semivolatiles	ENA TID	01,20,40, 16,46					
Characterization Volatiles	VOA CHR	01,20,40, 16,46					
Characterization Semivolatiles	ENA CHR	01,20,40, 16,46					
Direct Aqueous Injection Volatiles	VOA DAI	01					
Direct Aqueous Injection Semivolatiles	ENA DAI	01					

Bolded 01 & 20 - Std. Prd.

C - Change Noted

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 4.

Last Revision: 6/8/87 Current Revision: 9/15/87

<<< >>>		<<< >>>		INORGANIC CHEMISTRY <<< >>>		<<< >>>	
Physical Tests	Test ID	Matrices	A	B	C	D	E
@ Corrosivity By pH	METPH #	01,20					
Corrosivity, NACE	NACE	01,20					
@ Color	NESCOLR	01					
@ Odor	ODOR	01					
Particle Size / Hydrometer		20					
Particle Size / Sieve		20					
Ignitability, Closed Cup	FLSHPT	01,20,40					
Percent Oil/Water/Solid (O/W/S)	%OWS	40					
Percent O/W/S (Modified Oven Technique)	%OWSMOD	40					
Oil & Grease / Gravimetric	BAL O&G	01,20					
Oil & Grease / Infrared Spectrometer (IR)	IR O&G	01,20					
Aromatic Oil & Grease / (IR)	IR AO&G	01,20					
Total Petroleum Hydrocarbons (TPH) / (IR)	IR TPH	01,20					
Mineral Tests							
@ Specific Conductance	CELSC #	01,20					
@ Acidity	METACID	01,20					
@ pH	METPH #	01,20					
pH, Paste	METPHP	20					
@ Alkalinity, Total/Carb/Bicarb/Hydroxide	METALK	01,20					
Hardness, Titration	BURHARD ~~~~~						
Hardness, ICP	ICPHAR*						
Sodium Adsorption Ratio (SAR)	ICP SAR	01,20					
Cation Exchange Capacity	ICP CEC	20					
Ion Balance Calculation	IONBALCALC						
Ion Balance (Major Cations/Anions)	See Job Code: IONBALANCE						
Oxygen Demand / Carbon							
@ Biochemical Oxygen Demand (BOD)	METBOD	01					
Chemical Oxygen Demand (COD)	METCOD	01,20					
Total Organic Carbon (TOC)	TOCTOC#	01,(20 Div.12)					
Purgeable Organic Carbon (POC)	TOCPOC	01					
Dissolved Organic Carbon (DOC)	TOCDOC	01					

* S-Single & Q-Quad for 01 Matrix Only; Other matrices do not need an additional letter
 * T-Total, D-Dissolved, R-Recoverable.

@ SHORT HOLDING TIMES C-Change Noted.

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 5.

Last Revision: 06/08/87 Current Revision: 09/15/87

Nitrogen	Test ID	Matrices	A	B	C	D	E
Total Kjeldahl Nitrogen (TKN)	TECTKN	01,20					
<u>Ammonia, Nitrogen</u>	TECNH3	01,20					
Ammonia, Distilled	TECNH3T	01,20					
@ <u>Nitrite, Nitrogen</u>	TECNO2	01,20					
@ <u>Nitrate, Nitrogen</u>	TECNO3	01,20					
<u>Nitrite Plus Nitrate, Nitrogen</u>	TECNOXT	01,20					
@ <u>Nitrate, IC</u>	IC NO3	01,20					
@ <u>Nitrite, IC</u>	IC NO2	01,20					
Total Organic Nitrogen	See Job Code : TON01						
Phosphorus							
@ Orthophosphate, Colorimetric	TECO P	01,20					
@ Orthophosphate, IC	IC PO4	01,20					
Polyphosphate, IC	IC PPO4	01,20					
Total Phosphorus, Colorimetric	TECT P	01,20					
Phosphorus, ICP	See ICP Suite Compounds						
Solids							
Total Solids (TS)	BALTS	01,20					
Total Suspended (TSS)	BALTSS	01					
@ Total Dissolved Solids (TDS)	BALIDS	01					
Total Volatile Solids (TVS)	BALIVS	01,20					
Volatile Suspended Solids (VSS)	BALVSS	01					
@ Turbidity (NTU)	SPETURB	01					
Settleable Solids (SS)	CONESS	01					
Microbiology							
@ Coliform, Total	COLIF T	01					
@ Coliform, Fecal	COLIF F	01					

* S-Single & Quad for 01 Matrix Only; Other matrices do not need an additional letter
 * T-Total, D-Dissolved, R-Recoverable.
 C-Change noted.

@ SHORT HOLDING TIMES

Underlined Items Are Preferred

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 6.

Last Revision: 6/8/87 Current Revision: 9/15/87

Sulphur	Test ID	Matrices	A	B	C	D	E
<u>Sulfate, IC</u>	IC SO4	01,20					
Sulfate, Turbidimetric	SPES04	01,20					
@ Sulfite, Titrimetric	BURS03	01,20					
@ Sulfite, IC	IC SO3	01,20					
<u>Sulfide, Colorimetric</u>	SPES *	01,20					
Sulfide, IC	IC S	01,20					
Sulfur, ICP	See ICP Suite Compounds						
Sulfide - Reactive	SPES R	01,20					
Thiosulfate, IC	IC S2O3	01,20					
Thiocyanate, IC	IC SCN	01,20					
Cyanide							
Cyanide, Total	TEOCN T	01,20					
Cyanide, Amenable to Chlorination	TEOCN F	01,20					
Cyanide, Weak & Dissociable	TEOCN W	01,20					
Cyanide, IC	IC CN	01,20					
Cyanide - Reactive	TEOCN R	01,20					
Halogens							
Bromide, IC	IC BR	01,20					
Chloride, Titrimetric	BURCL	01,20					
<u>Chloride, IC</u>	IC CL	01,20					
@ Chlorine, Residual	POTCL2R	01,20					
Perchlorate, IC	IC ClO4	01,20					
<u>Fluoride, Electrode</u>	METF	01,20					
Fluoride, Distilled, Electrode	METF T	01,20					
<u>Fluoride, IC</u>	IC F	01,20					
Iodide, IC	IC I	01,20					
Total Organic Halogen (TOX)	TOXTOX*	01,20					
Purgeable Organic Halogen (POX)	TOXPOX	01					
Dissolved Organic Halogen (DOX)	TOXDOX	01					

* S-Single & Q-Quad for 01 matrix only; Other matrices do not need an additional letter

- T-Total, D-Dissolved, R-Recoverable.

@ SHORT HOLDING TIMES,

C-Change Noted.

Underlined Items Are Preferred

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

SOP No. LP-RMA-0004
Page: 9 of 29
Date: 12/9/87
Revision: Original

LONG-FORM pg 7.

Last Revision: 6/8/87 Current Revision: 9/15/87

Radiochemistry	Test ID	Matrices	A	B	C	D	E
Gross Alpha & Beta	RADA&B	01,20					
Lead 210	RAPB210	01,20					
Radium 226	RAD226	01,20					
Radium 228	RAD228	01,20					
Thorium 230	RATH230	01,20					
Uranium, Natural	TOXDOX	01,20					
Other Tests							
Tarmin / Lignin		01,20 Div.22					
Phenolics (4-AAP)	SPEPHEN	01,20					
@ Surfactants (MBAS)	SPEMBAS	01,20					
CLTENHOLD							
TCLP Master Preps							
TCLP Prep / EXTRACTABLE Organics Only**	M40TCLPO	40					
TCLP Prep / VOLATILE Organics Only	M40ZHE	40					
TCLP Prep / METALS Only	M40TCLPM	40					
TCLP Prep / METALS & EXTRACT. ORGS Only	M40TCLP	40					

** Includes Pesticides

S-Single & Q-Quad for 01 Matrix Only; Other matrices do not need an additional letter.
* D-Dissolved, T-Total, R-Recoverable;
@ SHORT HOLDING TIMES C-Change Noted.

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

SOP No. LP-RMA-0004
Page: 10 of 29
Date: 12/9/87
Revision: Original

LONG FORM pg 8.

Last Revision: 6/8/87 Current Revision: 9/15/87

Trace Metals by ICP & AA	Test ID	Matrices	A	B	C	D	E
* ICP Scan / 27 Metals, Standard Product	ICP LI*	01,20					
ICP Metals, Soluble Salts	ICP SS	01,20					
ICP Suite / Choose From List Below	ICP*	01,20,T16,T46	See below				

Choose: ICP Suite, AA Metals	Test ID	A	B	C	D	E	Choose: ICP Suite, AA Metals	Test ID	A	B	C	D	E
Aluminum, ICP							Manganese, ICP						
Antimony, Furn AA	FSB*						Mercury, CV AA	CVHG*					
Antimony, ICP							Molybdenum, ICP						
Arsenic, Furn AA	FAS*						Nickel, ICP						
Arsenic, Hyd Gen	DIV. 22						Osmium, ICP						
Arsenic, ICP							Phosphorus, ICP						
Barium, ICP							Potassium, ICP						
Beryllium, ICP							Selenium, ICP						
Boron, ICP							Selenium, Furn AA	FSE*					
Cadmium, Furn AA	FCD*						Selenium, Hyd Gen	Div. 22					
Cadmium, ICP							Silica (SiO2), ICP						
Calcium, ICP							Silicon, ICP						
Chromium (III)	CR+3 ₄						Silver, Furn AA	FAG*					
Chromium (VI)	SPECR6*						Silver, ICP						
Chromium, ICP							Sodium, ICP						
Cobalt, ICP							Strontium, ICP						
Copper, ICP							Sulphur						
Iron, ICP							Thallium, Furn	FTL*					
Lead, Tot Organic							Tin, ICP						
Lead, Furnace AA	FPB*						Titanium, ICP						
Lead, ICP							Uranium, Natural						
Lithium, ICP							Vanadium, ICP						
Magnesium, ICP							Zinc, ICP						

* D-Dissolved, T-Total, R-Recoverable; (01,20,16,46 matrices for ICP* & Furnace Tests)
♦ DIS - Dissolved, TOT- Total.

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

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Revision: Original

LONG FORM pg 9.

Last Revision: 6/8/87 Current Revision: 9/15/87

Inorganic Regulatory Packages	Job Code	Matrices	A	B	C	D	E
Appendix VIII Metals/Inorganics	AP8**MI	01,09,20,40,16					
Appendix IX Metals/Inorganics	AP9**MI	01,09,20,40,16					
Appendix IX Optional-Water Chem. Parameters	IONBALANCE	09					
Hazardous Substance List (HSL) Met/Inorg	HSL**MI	01,09,20,40,16					
CLP / HSL Metals/Inorganics	CLP**MI	01,09,20,40					
SDWA Primary Metals / Inorganics	SDWAP**M/I	01,09					
SDWA Secondary Metals / Inorganics	SDWAS**MI	01,09					
RCRA Total Metals	RCRA**M	01,09,20,40					
RCRA EP I Metals	EPI RCRM	Std. Prd. DL					
RCRA EP II Metals	EPII RCRM						
RCRA Groundwater Suitability	RCRAS**M/I/R	01,09					
RCRA Water Quality Metals/Inorganics	RCRAQ**MI	01,09					
RCRA Groundwater Quality Indicators	RCRAI**MI#	01,09					
Priority Pollutant Metals	PP**M	01,09,20,40,16					
Priority Pollutant Inorganics	PP**I	01,09,20,40					
Refinery Total Metals (Hazardous Constituent)	REFHC**M	01,09,20,40,16					
Refinery EP I Metals	EPI REFM	40					
Refinery EP II Metals	EPII REFM	40					
NPDES Part A Inorganics	NPDA**I	01					
NPDES Part B Metals / Inorganics / RAD	NPDB**MIR	01					
NPDES Part C Metals / Inorganics	PP**M/I	01,09,20,40					
TCLP Metals Aqueous Leachate	OTC**M	01,20,16					
TCLP Refinery Metals	See REFHC16M						

* S-Single, Q-Quad

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

S LONG FORM pg10.

[illegible]

This image shows a single sheet of white paper with horizontal ruling lines. The lines are evenly spaced and run across the width of the page. There is no text or other markings on the paper.

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

JOB CODES pg 1

Last Revision: 4/2/87 Issued: 6/8/87

SDWA Drinking Water Parameters	J.CODE ID	Matrices	A	B	C	D	E
Primary Complete	SDWAP**C	01,09					
Primary Metals	SDWAP**M	01,09					
Primary Inorganics	SDWAP**I	01,09					
Primary Radiochemistry	SDWAP**R	01,09					
Primary Organics	SDWAP**O	01,09					
Secondary Metals / Inorganics	SDWAS**MI	01,09					
Priority Pollutants							
Complete	PP**C	01,09,20,40					
Metals	PP**M	01,09,20,40,16					
Inorganics	PP**I	01,09,20,40					
Organics	PP**O	01,09,20,40,16					
Hazardous Substance List							
Complete	HSL**C	01,09,20,40					
Metals/Inorganics	HSL**MI	01,09,20,40,16					
Organics	HSL**O	01,09,20,40,16					
RCRA Groundwater - Monitoring Parameters							
Suitability Complete	RCRAS**C	01,09					
Suitability Metals	RCRAS**M	01,09					
Suitability Inorganics	RCRAS**I	01,09					
Suitability Radiochemistry	RCRAS**R	01,09					
Suitability Organics	RCRAS**O	01,09					
Quality Metals / Inorganics	RCRAQ**MI	01,09					
Indicator Inorganics	RCRAI**I#	01,09					
CLP / EPA Report Packages							
Complete	CLP C						
Metals / Inorganics	CLP MI						
Organics	CLP O						

S-Single, Q-Quad For 01 & 09 Matrices Only

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

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Revision: Original

JOB CODES pg 2.

Last Revision: 4/2/87 Issued: 6/8/87

Waste Characteristics & Other Tests	J.CODE ID	Matrices	A	B	C	D	E
WC Inorganics	RCRAWC40I	40					
EPI/ Metals	EPI RCRM						
EPI/ Organics	EPI RCRO						
EPII Oily Waste Metals	EPII RCRM						
TCLP Waste Characteristic (mark one: M/O/C)	OTC**M/O/C	01,20,16					
Land Restriction Rule	LRR**O	01,20,16					
RCRA Metals	RCRA**M	01,09,20,40,16					
Refinery Hazardous Constituents (HC)							
HC Complete	REFHC**C	01,09,20,40,16					
HC Metals	REFHC**M	01,09,20,40,16					
HC Organics	REFHC**O	01,09,20,40,16					
Waste Characteristics - Refinery							
Inorganics	REFWC**I	40					
EPI Metals	EPI REFM						
EPII Oily Waste Metals	EPII REFM						
Appendix 8 List							
Complete	AP8**C	01,09,20,40,16					
Metals / Inorganics	AP8**MI	01,09,20,40,16					
Organics	AP8**O	01,09,20,40,16					
Appendix 9 List							
Complete	AP9**C	01,09,20,40,16					
Metals / Inorganics	AP9**MI	01,09,20,40,16					
Organics	AP9**O	01,09,20,40,16					
Ionbalance							
Complete	IONBALANCE	01,09					
Cations	CATIONS	01,09					
Anions	ANIONS	01,09					

TCLP MASTER,(M40), PREPS : THESE ARE NO LONGER INCLUDED IN JOB CODES !!

YOU MUST PIECE THE M PREPS TOGETHER WITH DESIRED ANALYSES ; SEE FLOWCHARTS

TCLP Prep / EXTRACTABLE Organics Only *	M40TCLFO	40					
TCLP Prep / VOLATILE Organics Only	M40ZHE	40					
TCLP Prep / METALS Only	M40TCLFM	40					
TCLP Prep / METALS & EXTRACT. ORGS Only	M40TCLP	40					

* Includes Pesticides

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

INORG CHEMISTRY pg. 1. Last Revision: 6/8/87 Current Revision: 9/15/87				Job Code : Y N
Project #	Proj. Mgr.:	Prepared By:	Date: / /	Spcl. Inst: Y N
Group	Smpl Mtx	Test Mtx	Client Description	RMA Sample Numbers
A				
B				
C				
D				
E				

Hazard Label:

Physical Tests	Test ID	Matrices	A	B	C	D	E
@ Corrosivity by pH	METPH #	01,20					
Corrosivity, NACE	NACE	01,20					
@ Color	NESCOLR	01					
@ Odor	ODOR	01					
Particle Size / Hydrometer		20					
Particle Size / Sieve		20					
Ignitability, Closed Cup	FLSHPT	01,20,40					
Percent Oil / Water / Solids	%OWS	40					
Oil & Grease / Gravimetric	BAL O&G	01,20					
Oil & Grease / Infrared Spectrometer (IR)	IR O&G	01,20					
Aromatic Oil & Grease / IR	IR AO&G	01,20					
Total Petroleum Hydrocarbons (TPH) / IR	IR TPH	01,20					

Mineral Tests

@ Specific Conductance	CELSC #	01,20					
@ Acidity	METACID	01,20					
@ pH	METPH #	01,20					
pH, Paste	METPHP	20					
@ Alkalinity, Total/Carb/Bicarb/Hydroxide	METALK	01,20					
Hardness, Titration	BURHARD *****						
Hardness, ICP	ICPHAR*						
Sodium Adsorption Ratio (SAR)	ICP SAR	01,20					
Cation Exchange Capacity	ICP CEC	20					
Ion Balance Calculation	IONBALCALC						
Ion Balance (Major Cations/Anions)	See Job Code: IONBALANCE						

Oxygen Demand / Carbon

@ Biochemical Oxygen Demand (BOD)	METBOD	01					
Chemical Oxygen Demand (COD)	METCOD	01,20					
Total Organic Carbon (TOC)	TOCTOC#	01,(20 Div.12)					
Purgeable Organic Carbon (POC)	TOCPOC	01					
Dissolved Organic Carbon (DOC)	TOCDOC	01					

* S-Single & Quad for 01 Matrix Only; Other matrices do not need an additional letter
 ** T-Total, D-Dissolved, R-Recoverable.
 C-Change noted.

@ SHORT HOLDING TIMES

Figure 2 - Inorganic and Metal Analyses

INORGANIC CHEMISTRY pg 2.

Last Revision: 6/8/87 Current Revision: 9/15/87

Nitrogen	Test ID	Matrices	A	B	C	D	E
Total Kjedahl Nitrogen (TKN)	TECTKN	01,20					
Ammonia, Nitrogen	TECNH3	01,20					
Ammonia, Distilled	TECNH3T	01,20					
@ Nitrite, Nitrogen	TECNO2	01,20					
@ Nitrate, Nitrogen	TECNO3	01,20					
Nitrite Plus Nitrate, Nitrogen	TECNOKT	01,20					
@ Nitrate, IC	IC NO3	01,20					
@ Nitrite, IC	IC NO2	01,20					
Total Organic Nitrogen	See Job Code: TON01						
Phosphorus							
@ Orthophosphate, Colorimetric	TECO P	01,20					
@ Orthophosphate, IC	IC PO4	01,20					
Polyphosphate, IC	IC PPO4	01,20					
Total Phosphorus, Colorimetric	TECT P	01,20					
Phosphorus, ICP	See ICP Suite Compounds						
Solids							
Total Solids (TS)	BALTS	01,20					
Total Suspended Solids (TSS)	BALTSS	01					
Total Dissolved Solids (TDS)	BALIDS	01					
Total Volatile Solids (TVS)	BALTVS	01,20					
Volatile Suspended Solids (VSS)	BALVSS	01					
@ Turbidity (NTU)	SPEITURB	01					
Settleable Solids (SS)	CONESS	01					
Microbiology							
@ Coliform, Total	COLIF T	01					
@ Coliform, Fecal	COLIF F	01					
Sulphur							
Sulfate, IC	IC SO4	01,20					
Sulfate, Turbidimetric	SPESO4	01,20					
@ Sulfite, Titrimetric	BURSO3	01,20					
@ Sulfite, IC	IC SO3	01,20					
Sulfide, Colorimetric	SPES *	01,20					
Sulfur, ICP	See ICP Suite Compounds						
Sulfide - Reactive	SPES R	01,20					
Thiosulfate, IC	IC S2O3	01,20					
Thiocyanate, IC	IC SCN	01,20					

* T-Total, D-Dissolved; for 01 matrix;
Total sulfide only, for 20 matrix.

Underlined Items Are Preferred

Figure 2 - Inorganic and Metal Analyses

INORGANIC CHEMISTRY pg 3.

Last Revision: 6/8/87

Current Revision: 9/15/87

Cyanide	Test ID	Matrices	A	B	C	D	E
Cyanide, Total	TEOCN T	01,20					
Cyanide, Amenable to Chlorination	TEOCN F	01,20					
Cyanide, Weak & Dissociable	TEOCN W	01,20					
Cyanide, IC	IC CN	01,20					
Cyanide - Reactive	TEOCN R	01,20					
Halogens							
Bromide, IC	IC BR	01,20					
Chloride, Titrimetric	HURCL	01,20					
Chloride, IC	IC CL	01,20					
@ Chlorine, Residual	FOICL2R	01,20					
Perchlorate, IC	IC ClO4	01,20					
Fluoride, Electrode	METF	01,20					
Fluoride, Distilled, Electrode	METF T	01,20					
Fluoride, IC	IC F	01,20					
Iodide, IC	IC I	01,20					
Total Organic Halogen (TOX)	TOXTOX#	01,20					
Purgeable Organic Halogen (POX)	TOXPOX	01					
Dissolved Organic Halogen (DOX)	TOXDOX	01					
Radiochemistry							
Gross Alpha & Beta	RADA&B	01,20 Div.12					
Lead 210	RAPB210	01,20 Div.12					
Radium 226	RAD226	01,20 Div.12					
Radium 228	RAD228	01,20 Div.12					
Thorium 230	RATH230	01,20 Div.12					
Uranium, Natural	RADU	01,20 Div.12					
Other Tests							
@ Tannin / Lignin	DIV. 22	01,20					
Phenolics (4-AAP)	SPEPHEN	01,20					
@ Surfactants (MBAS)	SPEMBAS	01,20					
Major Anion Scan by Ion Chromatography	IC SCAN	01,20					
TCLP Master Prep							
TCLP Prep / METALS Only	MAOTCLPM	40					

* S-Single, Q-Quad, for 01 matrix only;
20 matrix leave blank.

Underlined Items Are Preferred

Figure 2 - Inorganic and Metal Analyses

INORGANIC CHEMISTRY pg 4.

Last Revision: 6/8/87 Current Revision: 9/15/87

Trace Metals by ICP & AA	Test ID	Matrices	A	B	C	D	E
ICP Scan / 27 Metals, Standard Product	ICP LI*	01,20,16					
ICP Metals , Soluble Salts	ICP SS	01,20					
ICP Suite / Choose from Lists Below	ICP*	01,20,T16,T46	See below				

Choose: ICP Suite, AA Metals	Test ID	A	B	C	D	E	Choose: ICP Suite, AA Metals	Test ID	A	B	C	D	E
Aluminum, ICP							Manganese, ICP						
Antimony, Furn AA	FSB*						Mercury, CV AA	CVHG*					
Antimony, ICP							Molybdenum, ICP						
Arsenic, Furn AA	FAS*						Nickel, ICP						
Arsenic, Hyd Gen	DIV. 22						Cadmium, ICP						
Arsenic, ICP							Phosphorus, ICP						
Barium, ICP							Potassium, ICP						
Beryllium, ICP							Selenium, ICP						
Boron, ICP							Selenium, Furn AA	FSE*					
Cadmium, Furn AA	FCD*						Selenium, Hyd Gen	DIV. 22					
Cadmium, ICP							Silica (SiO2), ICP						
Calcium, ICP							Silicon, ICP						
Chromium (III)	CR+3 ϕ						Silver, Furn AA	FAG*					
Chromium (VI)	SPECR6*						Silver, ICP						
Chromium, ICP							Sodium, ICP						
Cobalt, ICP							Strontium, ICP						
Copper, ICP							Sulphur						
Iron, ICP							Thallium, Furn	FTL*					
Lead, Tot Organic							Tin, ICP						
Lead, Furnace AA	FPB*						Titanium, ICP						
Lead, ICP							Uranium, Natural						
Lithium, ICP							Vanadium, ICP						
Magnesium, ICP							Zinc, ICP						

* D-Dissolved, T-Total, R-Recoverable; (01,20,16,46 matrix for ICP* and Furnace Tests)
 ϕ DIS - Dissolved, TOT- Total.

Figure 2 - Inorganic and Metal Analyses

INORGANIC CHEMISTRY pg 5.

Last Revision: 6/8/87 Current Revision: 9/15/87

Inorganic Regulatory Packages	Job Code	Matrices	A	B	C	D	E
Appendix VIII Metals / Inorganics	AP8**MI	01,09,20,40,16					
Appendix IX Metals / Inorganics	AP9**MI	01,09,20,40,16					
Appendix IX Optional-Water Chemistry Param	IONBALANCE	09					
Hazardous Substance List (HSL) Met / Inorg	HSL**MI	01,09,20,40,16					
CLP / HSL Metals / Inorganics	CLP**MI	01,09,20,40					
SDWA Primary Metals / Inorganics	SDWAP**M/I	01,09					
SDWA Secondary Metals / Inorganics	SDWAS**MI	01,09					
RCRA Total Metals	RCRA**M	01,09,20,40,16					
RCRA EP I Metals	EPI RCRM						
RCRA EP II Metals	EPII RCRM						
RCRA Groundwater Suitability	RCRAS**M/I/R	01,09					
RCRA Water Quality Metals/Inorganics	RCRAQ**MI	01,09					
RCRA Groundwater Indicators	RCRAI**MI#	01,09					
Priority Pollutant Metals	PP**M	01,09,20,40,16					
Priority Pollutant Inorganics	PP**I	01,09,20,40					
Refinery Total Metals (Hazardous Constituent)	REFHC**M	01,09,20,40,16					
Refinery EP I Metals	EPI REFM	40					
Refinery EP II Metals	EPII REFM	40					
NPDES Part A Inorganics	NPDA**I	01					
NPDES Part B Metals / Inorganics / RAD	NPDB**MIR	01					
NPDES Part C Metals / Inorganics	PP**M/I	01,09,20,40					
TCLP Metals Waste Characteristic Metals	OTC**M	01,20,16					
TCLP Refinery Metals	See REFHC**M						

Item Number	Changes

Comments For Sample Receiving:

S -Single, Q -Quad for 01 matrix only

Figure 2 - Inorganic and Metal Analyses

JOB CODES pg 1

Last Revision: 4/2/87 Issued: 6/8/87

SDWA Drinking Water Parameters	J.CODE ID	Matrices	A	B	C	D	E
Primary Complete	SDWAP**C	01,09					
Primary Metals	SDWAP**M	01,09					
Primary Inorganics	SDWAP**I	01,09					
Primary Radiochemistry	SDWAP**R	01,09					
Primary Organics	SDWAP**O	01,09					
Secondary Metals / Inorganics	SDWAS**MI	01,09					
Priority Pollutants							
Complete	PP**C	01,09,20,40					
Metals	PP**M	01,09,20,40,16					
Inorganics	PP**I	01,09,20,40					
Organics	PP**O	01,09,20,40,16					
Hazardous Substance List							
Complete	HSL**C	01,09,20,40					
Metals/Inorganics	HSL**MI	01,09,20,40,16					
Organics	HSL**O	01,09,20,40,16					
RCRA Groundwater - Monitoring Parameters							
Suitability Complete	RCRAS**C	01,09					
Suitability Metals	RCRAS**M	01,09					
Suitability Inorganics	RCRAS**I	01,09					
Suitability Radiochemistry	RCRAS**R	01,09					
Suitability Organics	RCRAS**O	01,09					
Quality Metals / Inorganics	RCRAQ**MI	01,09					
Indicator Inorganics	RCRAI**I#	01,09					
CLP / EPA Report Packages							
Complete	CLP C						
Metals / Inorganics	CLP MI						
Organics	CLP O						

* S-Single, Q-Quad For 01 & 09 Matrices Only

Figure 2 - Inorganic and Metal Analyses

JOB CODES pg 2.

Last Revision: 4/2/87 Issued: 6/8/87

Waste Characteristics & Other Tests	J.CODE ID	Matrices	A	B	C	D	E
WC Inorganics	RCRAWC40I	40					
EPI/ Metals	EPI RCRM						
EPI/ Organics	EPI RCRO						
EPII Oily Waste Metals	EPII RCRM						
TCLP Waste Characteristic (mark one: M/O/C)	OTC**M/O/C	01,20,16					
Land Restriction Rule	LRR**O	01,20,16					
RCRA Metals	RCRA**M	01,09,20,40,16					
Refinery Hazardous Constituents (HC)							
HC Complete	REFHC**C	01,09,20,40,16					
HC Metals	REFHC**M	01,09,20,40,16					
HC Organics	REFHC**O	01,09,20,40,16					
Waste Characteristics - Refinery							
Inorganics	REFWC**I	40					
EPI Metals	EPI REFM						
EPII Oily Waste Metals	EPII REFM						
Appendix 8 List							
Complete	AP8**C	01,09,20,40,16					
Metals / Inorganics	AP8**MI	01,09,20,40,16					
Organics	AP8**O	01,09,20,40,16					
Appendix 9 List							
Complete	AP9**C	01,09,20,40,16					
Metals / Inorganics	AP9**MI	01,09,20,40,16					
Organics	AP9**O	01,09,20,40,16					
Ionbalance							
Complete	IONBALANCE	01,09					
Cations	CATIONS	01,09					
Anions	ANIONS	01,09					

TCLP MASTER,(M40), PREPS : THESE ARE NO LONGER INCLUDED IN JOB CODES !!

YOU MUST PIECE THE M PREPS TOGETHER WITH DESIRED ANALYSES ; SEE FLOWCHARTS

TCLP Prep / EXTRACTABLE Organics Only *	M40TCLPO	40					
TCLP Prep / VOLATILE Organics Only	M40ZHE	40					
TCLP Prep / METALS Only	M40TCLFM	40					
TCLP Prep / METALS & EXTRACT. ORGS Only	M40TCLP	40					

* Includes Pesticides

Figure 2 - Inorganic and Metal Analyses

SOP No. LP-RMA-0004

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Date: 12/9/87

Revision: Original

ORGANIC CHEMISTRY pg 1

Last Revision: 4/2/87 Issued: 6/8/87

Job Code Y N

Project # Proj Mgr: Prepared By: Date: / / Spcl Inst: Y N

GROUP	Smpl Mtx	Test Mtx	Client Description	RMA Sample Numbers
A				
B				
C				
D				
E				

Hazard Label:

GC/LC Analyses	Test ID	Approved Test Matrices	A	B	C	D	E
* SDWA Trihalomethanes	THM SDW	01					
* Halogenated Volatile Organics **	601LI	01,20,16,46					
Halogenated VOA Orgs (LOW DETECTION LIMIT)	601LIL	20					
* Aromatic Volatile Organics **	602LI	01,20,16,46					
Aromatic VOA Orgs (LOW DETECTION LIMIT)	602LIL	20					
Benzene, Toluene, Ethylbenzene, Xylenes	602BTEX	01,20,16,46					
SDWA Volatiles	MCL SDW	01					
Acrolein & Acrylonitrile	603LI	01,20					
Phenols	604LI	01,20					
Benzidines	605LI	01,20					
Phthalate Esters	606LI	01,20					
Nitrosamines	607LI	01,20					
Organochlorine Pesticides/PCB's 608	OCP PP	01,20					
OCP's/PCB's 608 (LOW DETECTION LIMIT)	OCP PPL	20					
NPDES Organochlorine Pesticides/PCB's	OCP PP	01,20					
* HSL Organochlorine Pesticides/PCB's **	OCP HSL	01,20,16,46					
HSL OCP's/PCB's (LOW DETECTION LIMIT)	OCPHSL	20					
CLP/HSL Organochlorine Pesticides/PCB's	OCP CLP	01,20					
Appendix 8 or 9 Organochlorine Pest/PCB's	OCP AP9	01,20					
TCLP Characteristic Organochlorine Pests.	OCP TCLP	01,20,16,46					
* SDWA Organochlorine Pesticides	OCP SDW	01					
* RCRA Organochlorine Pesticides	OCP RCR	01,20					
PCB's	PCB	01,20,45					
Nitro-Aromatics & Cyclic Ketones	609LI	01,20					
Polynuclear Aromatic Hydrocarbons / 610	LC PNA	01,20,16,46					
Haloethers	611LI	01,20					
Chlorinated Hydrocarbons	612LI	01,20					
Organophosphate Pesticides **	OPP	01,20					
Appendix 8 or 9 Organophosphate Pesticides	OPP AP9	01,20					
Appendix 8 or 9 Herbicides	HRB AP9	01,20					

** Most Modifiable Test

C - Change Noted

* Preferred Standard Product (Bolded 01 & 20)

01 & 20 Bolded - Std.Prd. DL

Figure 3 - Chromotography Analyses

ORGANIC CHEMISTRY pg 2

Last Revision: 4/2/87 Issued: 6/8/87

GC/LC Analyses Cont.	Test ID	Matrices	A	B	C	D	E
TCLP Herbicides	HRBTCLP	01,20,16,46					
* SDWA Herbicides **	HRB SDW	01					
* RCRA Herbicides **	HRB RCR	01,20					
Triazines	619LI	01,20					
Carbamate & Urea Pesticides, HPLC	632LI	01,20					
Penta & Tetrachlorophenol	PCP	01,20					
Ethylene dibromide (EDB)	504LI	01,20					
Hydrocarbon Scan by FID	GC HYD	01,20					
Boiling Point Distribution By GC	GC BPD	01,20					
Water Miscible Solvents	GC DAI	01					
Semivolatiles by MSD	MSD BNA	01,20					
Volatiles by MSD	MSD VOA	01,20					
Semivolatiles by FID	GC BNA	01,20					
Base Neutrals by FID	GC BN	01,20					
Acids by FID	GC ACD	01,20					
Land Treatment Demonstration /HPLC	LC LTD	01,20,16,46					
CLIENTHOLD							

>>> Other <<<							
TCLP Prep / EXTRACTABLE ORGANICS Only	TCLPO	M40					
TCLP Prep / VOLATILES Only	ZHE	M40					

Item Number	Changes

Comments To Sample Receiving :

** Most Modifiable Test

C- Change noted

* Preferred Standard Product (01 & 20 Bolded)

Figure 3 - Chromotography Analyses

SOP No. LP-RMA-0004

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Date: 12/9/87

Revision: Original

JOB CODES pg 1

Last Revision: 4/2/87 Issued: 6/8/87

SDWA Drinking Water Parameters	J.CODE ID	Matrices	A	B	C	D	E
Primary Complete	SDWAP**C	01,09					
Primary Metals	SDWAP**M	01,09					
Primary Inorganics	SDWAP**I	01,09					
Primary Radiochemistry	SDWAP**R	01,09					
Primary Organics	SDWAP**O	01,09					
Secondary Metals / Inorganics	SDWAS**MI	01,09					
Priority Pollutants							
Complete	PP**C	01,09,20,40					
Metals	PP**M	01,09,20,40,16					
Inorganics	PP**I	01,09,20,40					
Organics	PP**O	01,09,20,40,16					
Hazardous Substance List							
Complete	HSL**C	01,09,20,40					
Metals/Inorganics	HSL**MI	01,09,20,40,16					
Organics	HSL**O	01,09,20,40,16					
RCRA Groundwater - Monitoring Parameters							
Suitability Complete	RCRAS**C	01,09					
Suitability Metals	RCRAS**M	01,09					
Suitability Inorganics	RCRAS**I	01,09					
Suitability Radiochemistry	RCRAS**R	01,09					
Suitability Organics	RCRAS**O	01,09					
Quality Metals / Inorganics	RCRAQ**MI	01,09					
Indicator Inorganics	RCRAI**I#	01,09					
CLP / EPA Report Packages							
Complete	CLP C						
Metals / Inorganics	CLP MI						
Organics	CLP O						

S-Single, Q-Quad For 01 & 09 Matrices Only

Figure 3 - Chromotography Analyses

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JOB CODES pg 2.

Last Revision: 4/2/87 Issued: 6/8/87

Waste Characteristics & Other Tests	J.CODE ID	Matrices	A	B	C	D	E
WC Inorganics	RCRAWC4OI	40					
EPI/ Metals	EPI RCRM						
EPI/ Organics	EPI RCRO						
EPII Oily Waste Metals	EPII RCRM						
TCIP Waste Characteristic (mark one: M/O/C)	OTC**M/O/C	01,20,16					
Land Restriction Rule	LRR**O	01,20,16					
RCRA Metals	RCRA**M	01,09,20,40,16					
Refinery Hazardous Constituents (HC)							
HC Complete	REFHC**C	01,09,20,40,16					
HC Metals	REFHC**M	01,09,20,40,16					
HC Organics	REFHC**O	01,09,20,40,16					
Waste Characteristics - Refinery							
Inorganics	REFWC**I	40					
EPI Metals	EPI REFM						
EPII Oily Waste Metals	EPII REFM						
Appendix 8 List							
Complete	AP8**C	01,09,20,40,16					
Metals / Inorganics	AP8**MI	01,09,20,40,16					
Organics	AP8**O	01,09,20,40,16					
Appendix 9 List							
Complete	AP9**C	01,09,20,40,16					
Metals / Inorganics	AP9**MI	01,09,20,40,16					
Organics	AP9**O	01,09,20,40,16					
Ionbalance							
Complete	IONBALANCE	01,09					
Cations	CATIONS	01,09					
Anions	ANIONS	01,09					

TCIP MASTER,(M40), PREPS : THESE ARE NO LONGER INCLUDED IN JOB CODES !!

YOU MUST PIECE THE M PREPS TOGETHER WITH DESIRED ANALYSES ; SEE FLOWCHARTS

TCIP Prep / EXTRACTABLE Organics Only *	M40TCIP0	40					
TCIP Prep / VOLATILE Organics Only	M40ZHE	40					
TCIP Prep / METALS Only	M40TCIPM	40					
TCIP Prep / METALS & EXTRACT. ORGS Only	M40TCIP	40					

* Includes Pesticides

Figure 3 - Chromotography Analyses

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ORGANIC CHEMISTRY pg 1

Issued: 01/07/87 Revised: 03/03/87

Job Code : Y N

Project #	Proj Mgr:	Prepared By:	Date: / /	Spcl Inst: Y N
GROUP	Smpl Mtx	Test Mtx	Client Description	RMA Sample Numbers
A				
B				
C				
D				
E				

Hazard Label:

GC / MS ANALYSES	Test ID	Approved Test Matrices	A	B	C	D	E
Priority Pollutant Volatiles	VOA 624	01,20,40 16,46					
Priority Pollutant Semivolatiles	ENA 625	01,20,40 16,46					
Priority Pollutant Acid Organics	ACD 625	01,20,40					
Priority Pollutant Base/Neutral Organics	EN 625	01,20,40					
* Hazardous Substance List Volatiles	VOA HSL	01,20,40 16,46					
* Hazardous Substance List Semivolatiles	ENA HSL	01,20,40 16,46					
Hazardous Substance List Acid Organics	ACD HSL	01,20,40					
Hazardous Substance List Base/Neutral Org	EN HSL	01,20,40					
CLP/HSL Volatiles (TID's Included)	VOA CLP	01,20					
CLP/HSL Semivolatiles (TID's Included)	ENA CLP	01,20					
NPDES Volatile Organics	VOA 624	01					
NPDES Semivolatile Organics	ENA 625	01					
Appendix 8 or 9 Volatiles	VOA AP9	01,20,40, 16,46					
Appendix 8 or 9 Semivolatiles	ENA AP9	01,20,40, 16,46					
Appendix 8 or 9 Chlorinated Dioxins & Furans	DXN AP9	Div. 22					
Appendix 8 TID Volatiles	VOATID8	01,20,40, 16,46					
Appendix 8 TID Semivolatiles	ENATID8	01,20,40, 16,46					
TCIP Waste Characteristic Volatiles	VOATCLP	01,20,40, 16,46					
TCIP / Waste Characteristic Semivolatiles	ENATCLP	01,20,40, 16,46					
TCIP / Land Restriction Volatiles	VOA LRR	01,20,40, 16,46					
TCIP / Land Restriction Semivolatiles	ENA LRR	01,20,40, 16,46					
Refinery Hazardous Constituents Volatiles	VOA REF	01,20,40, 16,46					
Refinery Hazardous Constituents ENA	ENA REF	01,20,40, 16,46					
Polynuclear Aromatic Hydrocarbons	EN PNA	01,20,40, 16,46					
Polynuclear Aromatic Hydrocarbons SIM	ENSIPNA	01,20,40, 16,46					
Tentative Identification Volatiles	VOA TID	01,20,40, 16,46					
Tentative Identification Semivolatiles	ENA TID	01,20,40, 16,46					
Characterization Volatiles	VOA CHR	01,20,40, 16,46					
Characterization Semivolatiles	ENA CHR	01,20,40, 16,46					

C - Change Noted

* Preferred Standard Product (01 & 20 Bolded)

Bolded 01 & 20 - Std. Prd.

Figure 4 - Mass Spectrometry Analyses

Revision: Original

Issued: 01/07/87 Revised: 03/03/87

>>> OTHER <<<						
TCLP Prep for Organics Only	TCLPO	M40				
TCLP Prep for Volatiles	ZHE	M40				
TCLP Prep for Organics & Metals	TCLP	M40				

[illegible]

Comments To Sample Receiving :

Figure 4 - Mass Spectrometry Analyses

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JOB CODES pg 1.

Issued: 03/03/87 Revised: 02/03/87

SDWA Drinking Water Parameters	J.CODE ID	Matrices	A	B	C	D	E
Primary Complete	SDWAP**C	01,09					
Primary Metals	SDWAP**M	01,09					
Primary Inorganics	SDWAP**I						
Primary Radiochemistry	SDWAP**R	01,09					
Primary Organics	SDWAP**O	01,09					
Secondary Metals / Inorganics	SDWAS**MI	01,09					
Priority Pollutants							
Complete	PP**C	01,09,20,40					
Metals	PP**M	01,09,20,40					
Inorganics	PP**I	01,09,20,40					
Organics	PP**O	01,09,20,40					
Hazardous Substance List							
Complete	HSL**C	01,09,20,40					
Metals/Inorganics	HSL**MI	01,09,20,40					
Organics	HSL**O	01,09,20,40					
CLP / EPA Report Packages							
Complete	CLP C						
Metals / Inorganics	CLP MI						
Organics	CLP O						
RCRA Groundwater - Monitoring Parameters							
Suitability Complete	RCRAS**C	01,09					
Suitability Metals	RCRAS**M	01,09					
Suitability Inorganics	RCRAS**I	01,09					
Suitability Radiochemistry	RCRAS**R	01,09					
Suitability Organics	RCRAS**O	01,09					
Quality Metals / Inorganics	RCRAQ**MI	01,09					
Indicator Inorganics	RCRAI**I#	01,09					
Waste Characteristics Tests							
WC Inorganics	RCRAWC40I	40					
EPI/ Metals	EPI RCRM						
EPI/ Organics	EPI RCRO						
EPII Oily Waste Metals	EPII RCRM						

S-Single, Q-Quad For 01 & 09 Matrices Only

Figure 4 - Mass Spectrometry Analyses

JOB CODES pg 2.

Issued: 03/03/87 Revised: 02/01/87

Refinery Hazardous Constituents (HC)	J.CODE ID	Matrices	A	B	C	D	E
HC Complete	REFHC**C	01,09,20,40					
HC Metals	REFHC**M	01,09,20,40					
HC Organics	REFHC**O	01,09,20,40					
Waste Characteristics - Refinery							
Inorganics	REFWC**I	40					
EPI Metals	EPI REFM						
EPII Oily Waste Metals	EPII REFM						
Appendix 8 List							
Complete	AP8**C	01,09,20,40					
Metals / Inorganics	AP8**MI	01,09,20,40					
Organics	AP8**O	01,09,20,40					
Appendix 9 List							
Complete	AP9**C	01,09,20,40					
Metals / Inorganics	AP9**MI	01,09,20,40					
Organics	AP9**O	01,09,20,40					
Ionbalance							
Complete	IONBALANCE	01,09					
Cations	CATIONS	01,09					
Anions	ANIONS	01,09					
TCLP - Refinery							
Complete	TCLPREF						
Metals	TCLPREFM						
Semivolatiles	TCLPREFNA						
Volatiles	TCLPREFVOA						
TCLP - Waste Characteristics 6/13/86 Federal Registry							
(Federal Register) Complete	TCLPC						
Metals	TCLPM						
Semivolatiles	TCLPENA						
Herbicides	TCLPHERB						
Pesticides	TCLPPEST						
TCLP - Land Restriction Rule							
Complete	TCLPIRRC						
TCLP Other							
601 List	TCLP601						
602 List	TCLP602						
PNA by 610	TCLPPNA						
Other							
RCRA Metals	RCRA**M	01,09,20,40					

Figure 4 - Mass Spectrometry Analyses

STANDARD
OPERATING
PROCEDURE

Subject or Title: SAMPLE RECEIPT AND CHAIN OF CUSTODY - REFER TO QAPP SECTION 7.1 Page 1 of 7

SOP No.:
LP-RMA-0005

Revision No.:
Original

Effective Date:
12/9/87

Supersedes:

1. Purpose:

To document receipt of all samples to the laboratory. To notify lab personnel of all incoming samples. To notify lab personnel of arriving samples that contain short holding parameters. To record the transfer of samples from the client to the lab.

2. Policies:

Always assign a project number to every group of samples that arrive at the lab regardless of whether work is proceeded on them or not.

Project numbers are assigned in numerical order. USGS and MKE samples receive separate series of numbers. MKE samples require special chain of custody tracking.

3. Safety:

Always wear gloves and glasses while unpacking coolers. Coolers containing strong smelling samples must be unpacked under the hood area.

4. Procedure:

- a. As samples arrive they are given a unique project number for each group of samples from one client and recorded in the log book (Figure 1).
- b. Fill out the Sample checklist (Figure 2) while unpacking the samples.
- c. For samples arriving by a courier check that the custody seals are intact.
- d. Open the coolers, unpack the samples and check the information written on the chain of custody against what was received. Note any discrepancies such as missing samples, or broken bottles on the chain of custody form.
- e. Label all the samples (usually by sampling sites) with a project number and unique sample number (1,2,3,etc.). Record these numbers on the chain of custody next to the client identifications.

Prepared by:

Robert Kelly

Date:

12/10/87

Management Approval:

John W. Kelly

Date:

12/10/87

QA Officer Approval:

Robert C. Hamisch

Date:

12/19/87

SOP No.:
LP-RMA-0005Revision No.:
OriginalEffective Date:
12/9/87

- f. Sign and date the Chain of Custody (Figure 3). For samples hand delivered have the client sign and relinquish the custody. Always retain the top copy with the samples and only give a bottom copy to the client.
- g. Look for any inorganic short holding parameters and sign in these samples on the inorganic short holding clipboard (Figure 4). Look for any volatile parameters and sign these samples in on the Volatile clipboard (Figure 5).
- h. Take a picture of the samples. Label a manila file folder with the project number. Place the picture, checklist, chain of custody and any paperwork received in the folder.
- i. Deliver the file folder to the appropriate project manager.
- j. Place the samples in boxes and store in the walk in cooler on special shelves pending log in. Bottles needed to analyze the short holding parameters are placed in a special location in the walk in cooler.

5. Responsibilities:

Sample receiving personnel are responsible for signing the chain of custody upon receipt of samples, for knowing the location of the samples except when used by an analyst, and for signing out maximum security samples. Sample receiving personnel are responsible for noting the short holding parameters only when indicated on the paperwork from the client. Client managers must notify sample receiving if others are to be included.

6. Comments:

For maximum security of samples (beyond the storage in the secured facility) an internal chain of custody is provided. Analysts must sign for the samples in a special book and sign them in on return. The samples are stored in one of 3 locked refrigerators.

Revision: Original

[illegible]

Figure 1 - Log Book Record

PROJECT #

--	--	--	--	--

DIV:

--	--	--	--

COMPANY NAME: _____

SAMPLE CHECKLIST DONE BY: _____

____ COOLER(S) # _____ (RMA/CLIENT)

PICTURE TAKEN:

SEALS INTACT:

COOLER TEMP OK:

BOTTLES BROKEN OR LEAKING:

CONTAINERS LABELED:

RADIATION DETECTION:

CHAIN OF CUSTODY:

CC AGREES WITH SAMPLES:

VOA SAMPLES FILLED COMPLETELY:

SEDIMENT PRESENT IN WATERS:

SAMPLE CORRECTLY PRESERVED:

SHORT HOLDING TIMES:

() MS () VOA () 602 () IN

SAMPLE MATRIX: () WATER () SOIL () WASTE

OTHER: _____

TYPE OF BOTTLES: () RMA () CLIENT

DISCREPANCES:

Y	N

Figure 2 - Sample Checklist

4955 Yarrow Street, Arvada, CO 80002 (303) 421-6611

**A DIVISION OF
ENSECO
INCORPORATED**

RMAL Client _____

RMAL Project No. _____

Sampling Co. _____

Sampling Personnel _____

Project Name/No. _____

Sampling Site _____

[illegible]

Yellow Copy to Sampler

SS - 001

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Figure 3 - Sample Chain of Custody Record

INORGANIC SHORT HOLDING TIME PARAMETERS RECEIVED!

[illegible]

Figure 4 - Inorganic Short Holding Clipboard

DATE:

Figure 5 - Volatile Clipboard

APPENDIX B

METHOD DETECTION LIMIT STUDIES/STANDARD OPERATING PROCEDURES

QUALITY ASSURANCE BRANCH

APR 05 1988

ENVIRONMENT SERVICES DIVISION

INDEX OF APPENDIX B

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3	Standard Operating Procedure - Part Per Trillion PNAs	19
4	Standard Operating Procedure - Part Per Billion PNAs	64
5	Standard Operating Procedure - Total Phenolics	9

Section 1
Method Detection Limit Study
PNA

Table 1. PNA Detection Limit Determination

Compound	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Sample #7	Standard Deviation (s)	Method Detection Limit (3s) *
2,3-Benzofuran	19.4*	20.9*	18.0*	19.5*	20.3*	21.5*	18.6*	1.70*	5.1*
2,3-Dihydroindene	4.3	4.2	4.7	3.7	3.8	4.9	4.7	0.46	1.4
1H-Indene	4.4	4.2	4.6	3.9	4.1	4.7	4.6	0.30	0.9
Naphthalene	20.5*	21.0*	18.5*	20.3*	23.0*	23.5*	17.6*	2.15*	6.5*
Benzo(B)thiophene	3.6	3.5	3.9	3.4	3.3	3.8	4.1	0.29	0.9
Quinoline	4.7	4.0	4.1	3.7	3.3	4.4	4.1	0.45	1.4
1H-Indole	3.7	4.5	5.6	3.2	3.2	4.2	4.0	0.84	2.5
2-Methylnaphthalene	5.4	5.0	5.3	5.1	4.8	4.9	5.7	0.31	0.9
1-Methylnaphthalene	4.5	4.2	4.6	3.8	3.7	4.7	5.2	0.53	1.6
Biphenyl	17.9*	18.1*	16.4*	18.4*	18.1*	19.3*	15.0*	1.43*	4.3*
Acenaphthylene	3.9	3.6	4.6	3.7	3.5	4.4	4.5	0.46	1.4
Acenaphthene	4.2	3.7	4.7	3.5	3.5	4.1	4.1	0.43	1.3
Dibenzofuran	4.3	3.9	4.6	4.1	3.7	4.6	4.2	0.34	1.0
Fluorene	4.4	4.0	4.5	4.0	4.0	4.6	4.8	0.33	1.0
Dibenzothiophene	4.0	3.5	4.0	3.5	3.2	3.9	4.2	0.36	1.1
Phenanthrene	4.7	3.9	4.7	3.9	3.6	4.2	4.5	0.43	1.3
Anthracene	4.5	3.8	4.5	4.1	3.6	4.1	4.6	0.38	1.1
Acridine	4.1	4.3	4.9	4.1	3.8	2.4	2.3	0.98	2.9
Carbazole	4.5	3.2	4.8	3.5	3.9	3.1	3.8	0.64	1.9
Fluoranthene	4.5	3.8	4.7	3.9	3.6	4.4	4.7	0.45	1.4
Pyrene	4.3	3.7	4.4	3.9	3.4	4.2	4.7	0.45	1.4
Benzo(A)anthracene	4.6	3.6	4.0	3.6	3.3	5.3	5.3	0.83	2.5
Chrysene	4.3	3.3	3.7	3.3	2.9	5.1	5.3	0.94	2.8
Benzo(B)fluoranthrene	4.6	3.4	3.8	3.6	2.8	4.9	5.0	0.83	2.5
Benzo(K)fluoranthrene	4.1	3.2	3.5	3.2	3.2	4.9	4.8	0.76	2.3
7,12-Dimethylbenzanthracene	5.3	3.9	5.5	5.3	4.3	6.0	6.6	0.93	2.8
Benzo(E)pyrene	4.9	3.8	4.1	3.3	3.5	4.9	4.4	0.64	1.9
Benzo(A)pyrene	4.5	3.2	3.8	3.2	2.9	4.8	4.5	0.76	2.3
Perylene	4.6	3.6	3.8	3.5	3.3	5.3	5.1	0.82	2.5
3-Methylcholanthrene	4.3	4.1	3.9	3.4	3.2	4.9	6.7	1.18	3.5
Indeno(1,2,3-CD)pyrene	4.5	3.4	3.4	2.9	3.0	4.5	4.2	0.69	2.1
Dibenz(A,C)anthracene **	4.2	3.5	3.6	3.1	3.3	4.6	4.1	0.54	1.6
Dibenz(A,H)anthracene **	4.2	3.5	3.6	3.1	3.3	4.6	4.1	0.54	1.6
Benzo(G,H,I)perylene	3.8	3.0	2.9	2.6	2.9	4.9	4.7	0.94	2.8

Note: Amount spiked = 5 ng/L.

* Data for 2,3-Benzofuran, Naphthalene and Biphenyl were obtained from previous detection limit study. Spike levels = 20 ng/L.

** Compounds co-elute

Table 2. † Recoveries

Compound	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Sample #7	Method Blank	Recovery Range	Average Recovery
2,3-Benzofuran	97*	104*	90*	98*	101*	100*	83*	N/D	83-108*	97*
2,3-Dihydroindene	87	84	94	75	77	98	94	N/D	75-98	87
1H-Indene	88	84	92	79	82	94	92	N/D	79-94	87
Naphthalene	103*	105*	93*	101*	115*	118*	88*	N/D	88-118*	103*
Benzo(B)thiophene	72	70	77	68	66	76	81	N/D	66-81	73
Quinoline	93	79	82	74	68	87	83	N/D	66-93	81
1H-Indole	73	89	111	63	64	85	81	N/D	63-111	81
2-Methylnaphthalene	109	101	107	101	97	99	115	N/D	97-115	104
1-Methylnaphthalene	90	84	93	76	74	94	104	N/D	74-104	88
Biphenyl	89*	91*	82*	92*	91*	96*	75*	N/D	75-96*	88*
Acenaphthylene	79	72	93	74	70	89	90	N/D	70-93	81
Acenaphthene	84	75	95	70	69	83	81	N/D	69-95	80
Dibenzofuran	87	77	91	82	74	93	83	N/D	74-93	84
Fluorene	87	81	91	81	79	92	96	N/D	79-96	87
Dibenzothiophene	80	70	80	69	65	78	84	N/D	65-84	75
Phenanthrene	94	77	94	79	73	85	90	N/D	73-94	84
Anthracene	91	76	90	81	72	82	91	N/D	72-91	83
Acridine	83	85	97	82	75	49	46	N/D	46-97	74
Carbazole	91	63	97	70	78	63	76	N/D	63-97	77
Fluoranthene	90	77	93	79	72	89	94	N/D	72-94	85
Pyrene	87	75	88	78	68	83	94	N/D	68-94	82
Benzo(A)anthracene	92	71	79	72	65	107	105	N/D	65-107	84
Chrysene	86	66	73	65	59	103	106	N/D	59-106	80
Benzo(B)fluoranthrene	92	67	76	73	57	97	100	N/D	57-100	80
Benzo(K)fluoranthrene	82	65	69	64	65	99	95	N/D	64-99	77
7,12-Dimethylbenzanthracene	106	77	110	106	85	121	132	N/D	77-132	105
Benzo(E)pyrene	98	76	81	66	69	99	88	N/D	66-99	82
Benzo(A)pyrene	89	64	75	64	57	96	89	N/D	57-96	76
Perylene	92	72	75	69	65	105	103	N/D	65-105	83
3-Methylcholanthrene	87	83	79	68	64	98	134	N/D	64-134	87
Indeno(1,2,3-CD)pyrene	90	68	68	57	61	91	84	N/D	57-91	74
Dibenz(A,C)anthracene	83	69	72	63	66	92	81	N/D	63-92	75
Dibenz(A,H)anthracene	83	69	72	63	66	92	82	N/D	63-92	75
Benzo(G,H,I)perylene	76	61	58	52	58	99	94	N/D	52-99	71
Naphthalene-d8 **	75	67	79	68	66	88	86	81	66-88	76
Fluorene-d10 **	91	78	94	82	80	100	100	93	78-100	89
Chrysene-d12 **	95	68	76	71	66	117	111	82	66-117	86

* Data for 2,3-Benzofuran, Naphthalene and Biphenyl were obtained from previous detection limit study. Spike levels = 20 ng/L.

** Surrogate compound.

N/D = Not detected.

Table 3: Low Level PNA Spike Results

Compound	Sample #1	Sample #2	Sample #3	Percent Recovery		
				Sample #1	Sample #2	Sample #3
2,3-Benzofuran	2.5	2.3	2.6	101	90	105
2,3-Dihydroindene	2.9	2.6	3.0	115	105	119
1H-Indene	2.2	2.2	2.7	89	89	109
Naphthalene	5.9*	N/A	N/A	118	N/A	N/A
Benzo(B)thiophene	1.7	1.9	1.8	68	74	73
Quinoline	2.5	2.2	2.0	101	89	81
1H-Indole	1.5	2.2	2.7	60	90	107
2-Methylnaphthalene	2.3	2.5	2.7	92	99	107
1-Methylnaphthalene	2.2	2.3	2.5	88	91	99
Biphenyl	4.2*	N/A	N/A	83	N/A	N/A
Acenaphthylene	2.0	1.9	2.1	79	76	83
Acenaphthene	2.3	2.5	2.4	91	100	95
Dibenzofuran	1.6	1.6	1.5	64	62	61
Fluorene	2.0	2.1	2.3	82	82	93
Dibenzothiophene	1.7	1.7	1.8	67	69	72
Phenanthrene	1.9	2.1	2.5	75	82	100
Anthracene	1.6	1.7	2.4	65	67	96
Acridine	0.9	1.0	1.2	37	41	47
Carbazole	1.3	1.3	1.3	50	53	53
Fluoranthene	1.9	2.5	2.4	75	101	96
Pyrene	1.9	3.2	2.4	77	128	97
Benzo(A)anthracene	2.5	2.5	2.8	100	101	113
Chrysene	2.4	2.2	2.6	95	90	103
Benzo(B)fluoranthrene	1.8	1.8	2.3	71	72	93
Benzo(K)fluoranthrene	2.2	2.2	2.3	89	89	90
7,12-Dimethylbenzanthracene	3.3	3.6	3.3	132	142	133
Benzo(E)pyrene	1.9	1.8	2.0	75	73	80
Benzo(A)pyrene	1.9	2.0	2.4	78	79	96
Perylene	2.2	2.1	2.6	89	82	102
3-Methylcholanthrene	2.2	2.3	2.1	88	90	82
Indeno(1,2,3-CD)pyrene	2.0	1.9	2.1	82	75	84
Dibenz(A,C)anthracene	1.7	1.8	2.0	68	72	80
Dibenz(A,H)anthracene	1.7	1.8	2.0	67	72	80
Benzo(G,H,I)perylene	2.2	2.1	2.3	90	85	91

Note: All compounds spiked at 2.5 ng/L.

* Data for Naphthalene and Biphenyl were obtained from previous study.

Spike levels = 5.0 ng/L.

N/A = Not applicable.

Section 2
Method Detection Limit Study
Total Phenolics

Total Phenolics Method Detection Limit Study

Enseco

<u>Sample #</u>	<u>Concentration Detected (mg/L)</u>
1	0.0315
2	0.0340
3	0.0291
4	0.0315
5	0.0291
6	0.0291
7	0.0315

Calculated Standard Deviation = 0.0018

**Calculated Method Detection Limit = 0.00579 mg/L
= 5.8 ug/L**

Section 3
Standard Operating Procedure
part-per-trillion PNAs

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Title: DETERMINATION OF LOW LEVEL (PART PER TRILLION)

PAH AND HETEROCYCLES IN WATER

1.0 Summary of the Method

This method has been designed for the analysis of polynuclear aromatic hydrocarbons (PAH) and heterocyclic compounds at the part per trillion level (ppt,ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by gas chromatography mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). The compounds listed in Table 1 can be quantitatively determined using this analytical method.

Four 1-liter volumes of sample are separated into two 2-liter samples and extracted with methylene chloride. Analysis of the combined and concentrated extract is performed by gas chromatography/mass spectrometry using the selected ion monitoring scanning mode under electron impact ionization conditions.

2.0 Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

3.0 Apparatus and Materials

3.1 Glassware

Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used

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in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, then methanol.

Glassware should then be oven dried at 150°C for 30 minutes, and heated in a muffle furnace at 400°C for 15 to 30 minutes. Solvent rinses with methylene chloride may be substituted for the muffle furnace heating. Volumetric glassware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants.

Store glassware inverted or capped with aluminum foil. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.1.1 Separatory funnel - 3000 mL, with Teflon stopcock.

3.1.2 Drying column - glass funnel with ~10 cm anhydrous sodium sulfate.

3.1.3 Concentrator tube, Kuderna-Danish - 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground-glass stoppers are used to prevent evaporation of extracts.

3.1.4 Snyder column, Kuderna-Danish - Three-ball macro (Kontes K-503000-0121 or equivalent).

3.1.5 Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs or clips.

3.1.6 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C. The N-Evap by Organomation Associates, Inc., South Berlin, MA (or equivalent) is suitable.

3.1.7 Micro reaction vessels, 2.0 mL (Supelco 3-3295).

3.2 Gas Chromatograph

The analytical system includes a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

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3.3 Column

A J&W 15 meter fused silica capillary column coated with DB-5 bonded phase, or equivalent.

3.4 Mass Spectrometer

A mass spectrometer operating at 70 ev (nominal) electron energy in the electron impact ionization mode and tuned to maximize the sensitivity of the instrument to the compounds being analyzed. The GC capillary column is fed directly into the ion source of the mass spectrometer.

A computer system interfaced to the mass spectrometer allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

4.0 Reagents

4.1 Reagent water

Reagent water is defined as water in which the target compounds are not observed at or above the method detection limit.

4.2 Solvents

Acetone, methanol, methylene chloride, cyclohexane - Burdick & Jackson, distilled in glass, or equivalent.

4.3 Sodium sulfate

(ACS) Granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray.

4.4 Surrogate Spiking Solution

A solution containing 10 ng/mL of each surrogate compound is prepared by weighing appropriate aliquots of the purified crystals into a volumetric flask and diluting to volume with methanol or acetone.

Two milliliters of the surrogate solution is added to each 2L aliquot of sample to give a sample concentration of X 10 ng/L.

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4.5 Internal Standard Solutions

A solution containing ca. 800 ng/mL of each internal standard is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methylene chloride. Fifty microliters of this solution is added to the extract (0.5 mL) prior to analysis to give a concentration of the internal standards in the extract of 80 ng/mL (equivalent to 10 ng/L in a 4L sample).

4.6 Matrix Recovery Standard Spiking Solution

A solution containing the following compounds at the listed concentrations is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methanol or acetone. The corresponding sample concentrations for each compound using the spiking protocol described in 6.3 are shown below:

<u>Compound</u>	<u>Spiking Solution Concentration (ng/ml)</u>	<u>Sample Concentration (ng/L)</u>
Naphthalene	20	20
Fluorene	20	20
Chrysene	20	20
Indene	20	20
Quinoline	20	20
Benzo(e)pyrene	20	20
2-methyl naphthalene	20	20

5.0 Sample Preservation, Storage and Holding Times

5.1 Sample Preservation and Storage

The samples must be protected from light and refrigerated at 4°C (+ 2°C) from the time of receipt until extraction and analysis. After analysis, extracts and unused sample volume must be protected from light and refrigerated at 4°C (+ 2°C).

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5.2 Holding Times

Samples must be extracted within 5 days of the time of sample receipt. Two days are allowed to ship samples from the field to the laboratory. The total time allowed from sample collection until extraction is therefore 7 days.

Extracts must be analyzed within 40 days of extraction.

6.0 Sample Extraction

6.1 Samples

Samples are extracted at $\text{pH} > 12$. Each 4-liter sample is separated into two 2-liter aliquots in two 3-liter separatory funnel with the surrogate spiking solution. A 2.00 mL volume of surrogate spiking standard is added to each 3-liter separatory funnel, to give a concentration of 10 ng/L (10ppt) of each surrogate. Each aliquot is then extracted three times (80 mL/80 mL/80mL) with methylene chloride. The three methylene chloride extracts are passed through an anhydrous sodium sulfate drying column, and combined in a Kuderna-Danish evaporative concentrator.

The extract is concentrated to approximately 0.5 mL and transferred to a 2.0 mL microreaction vessel. The methylene chloride is evaporated using a nitrogen stream. The evaporative concentrator tube is successively rinsed with methylene chloride, the rinsings added to the reaction vessel and the methylene chloride again evaporated. This process is continued until at least five (5) 1 mL rinsings of the tube have occurred.

The final methylene chloride extract is evaporated to 500 μL . All microreaction vessels are permanently marked at the 500 μL level and additional methylene chloride added, when necessary, to insure a final 500 μL extract volume. The extract vessel is capped with a Teflon fitted septum cap and stored at 4°C prior to GC/MS analysis.

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6.2 Method blank

Method blanks are prepared by treating a 4-L sample of laboratory reagent water exactly as described above. A method blank must be performed once each case*, each 14 calendar day period during which samples in a case are received, with every 20 samples of similar concentration and/or sample matrix or whenever samples are extracted by the same procedure, whichever is most frequent.

* A case is a group or a set of samples collected from a particular site over a given period of time.

6.3 Matrix Recovery Sample

Matrix recovery samples are prepared by spiking 2.00 mL of the matrix recovery standard spiking solution into two 2-L volumes of water for analyses. This gives a concentration of each matrix spike compound of 20 ng/L in the 4L water sample. The fortified sample is extracted exactly as described above for samples. The laboratory will spike and analyze 5% matrix spike samples (i.e. one matrix spike with every 20 samples).

6.4 Duplicate Sample

For a minimum of 10% of the samples analyzed a duplicate sample will be taken at sampling and a duplicate analysis will be performed. This will be carried out to insure that an estimate of precision will be available.

7.0 GC/MS Calibrations

Prior to use of the method for low level analysis of PAH, a five-point response factor calibration curve must be established showing the linear range of the analysis. The concentrations of standards used to construct the calibration curve are 40, 80, 200, 400, and 800 ng/mL. These concentrations correspond to 5, 10, 25, 50 and 100 ng/L in the samples if a 4L sample is extracted and the extract is concentrated down to 0.5 mL. If the concentration of any target compound in a sample exceeds the linear range defined by the standards above, the extract must be diluted so that the concentrations of all target compounds fall within the range of

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the calibration curve. For every 12 hours of GC/MS analysis, the mass spectrometer response for each PAH relative to the internal standard is determined, as described in the Calculations section, using daily check standards at concentrations of 80 ng/mL (10 ng/L in sample). Daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within ± 35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within ± 35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

Qualitative identification of target compounds will follow the relative retention time (RRT) criteria. Table 2 contains example RRT data for these compounds.

8.0 Daily GC/MS Performance Tests

The GC/MS will not be tuned to meet decafluorotriphenylphosphine (DFTPP) ion abundance criteria. EPA has dropped this requirement for selected ion monitoring (SIM) methods. This allows the laboratory to tune the instrument to maximize the sensitivity for the compounds being analyzed.

9.0 Gas Chromatography/Mass Spectrometry Analysis

Just prior to analysis an aliquot of internal standard solution is transferred to the sample vial using a 250 μ L syringe to give a final internal standard concentration of 80 ng/mL in the extract. Representative aliquots are injected into the capillary column of the gas chromatograph using the following, or similar conditions:

Injector Temp - 290°C
Transfer Line Temp - 310°C
Initial Oven Temp - 35°C
Initial Hold Time - 2 min.
Ramp Rate - 10°C/min.
Final Temperature - 310°C

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses for each PAH as shown in Table 1. Table 3 contains the SIM sequences used. For all compounds detected at a concentration above the MDL, a check is made to insure the confirmation ion is present.

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10.0 Calculations

10.1 Qualitative Identification

Obtain EICPs for the primary m/z and the confirmatory ion. The following criteria must be met to make a qualitative identification:

The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

The retention time must fall within ± 30 s of the retention time of the authentic compound.

The relative peak heights of the characteristic masses in the EICPs must fall within $\pm 20\%$ of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library.

Structural isomers that have very similar mass spectra and less than 30 s difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.2 Quantitation

The following formula is used to calculate the response factors of the internal standard to each of the calibration standards.

$$RF = (A_S C_{IS}) / (A_{IS} C_S)$$

where:

A_S = Area of the characteristic ion for the parameter to be measured.

A_{IS} = Area of the characteristic ion for the internal standard.

C_{IS} = Concentration of the internal standard, (ng/mL).

C_S = Concentration of the parameter to be measured, (ng/mL).

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Based on these response factors, sample extract concentrations for each PAH is calculated using the following formula.

$$C_e = \frac{(A_s)(I_s)}{(A_{is})(RF)}$$

where:

C_e = Sample extract concentration (ng/mL)

A_s = Area of the characteristic ion for the parameter to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

I_s = Amount of internal standard added to each extract (ng/mL).

The actual sample concentration (C) for each compound is calculated by the following formula:

$$C = (C_e) \times \sqrt{\frac{V_E}{V_S}}$$

C = Concentration in Sample (ng/L)

V_E = The final extract volume (mL), and

V_S = The original volume of sample extracted (L).

11.0 Quality Control/Quality Assurance

11.1 GC/MS Tuning

The GC/MS will not be tuned to meet decafluorotriphenylphosphine (DFTPP) ion abundance criteria. EPA has dropped this requirement for selected ion monitoring (SIM) methods. This allows the laboratory to tune the instrument to maximize the sensitivity for the compounds being analyzed.

11.2 GC/MS Initial Calibration and Continuing Calibration Check

Prior to the use of the method for low level analysis of PAH, a five-point response factor calibration curve must be established showing the linear range of the analysis.

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Each calibration standard is analyzed and the area of the primary characteristic ion is tabulated against concentration for each compound. The response factor (RF) for each compound at each concentration level is calculated using the following equation:

$$RF = \frac{A_s}{A_{is}} \times \frac{C_{is}}{C_s}$$

- A_s = Area of the characteristic ion for the compound to be measured.
 A_{is} = Area of the characteristic ion for the specific internal standard.
 C_{is} = Concentration of the internal standard
 C_s = Concentration of the compound to be measured.

For every 12 hours of GC/MS analysis, the mass spectrometer response (RF) for each PAH of interest (Table 1) relative to the internal standard is determined.

These daily response factors for each compound must be compared to the initial calibration curve. The percent difference is calculated using the following equation:

$$\% \text{ Difference} = \frac{\overline{RFI} - RFC}{\overline{RFI}} \times 100$$

\overline{RFI} = Average response factor from initial calibration.

RFC = Response factor from current verification check standard.

If the daily response factor are within ± 35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within ± 35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

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11.3 Method Blank Analysis

A method blank consists of deionized, distilled laboratory water carried through the entire analytical scheme (extraction, concentration, and analysis). The method blank volume must be approximately equal to the sample volumes being processed.

Method blank analysis are performed at the rate of one per case*, each 14 calendar day period during which samples in a case are received, with every 20 samples of similar concentration and/or sample matrix, or whenever samples are extracted by the same procedure, whichever is most frequent.

An acceptable method blank analysis must not contain any target compound in Table 1 at concentrations greater than or equal to the Method Detection Limits (MDL). If the method blank does not meet these criteria, the analytical system is out of control and the source of the contamination must be investigated and corrective measures taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control must be reextracted and reanalyzed if sufficient sample is available.

* A case is a group or a set of samples collected from a particular site over a given period of time.

11.4 Surrogate Compound Analysis

The laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compounds will be spiked into the sample prior to extraction and this will measure individual sample matrix effects associated with sample preparation and analysis. They will include naphthalene-d₈ fluorene d₁₀ and chrysene-d₁₂, at a sample concentration level of 10 ng/L (ppt).

A sample will be invalid for quantitative use in this program only if the recovery of any one or more of the surrogates falls outside the acceptance criteria. The initial acceptance criteria used for this program are the criteria established by ERT for these surrogates during 1986. The surrogate recovery acceptance criteria will be updated quarterly. RMAL will take corrective action whenever the surrogate recovery for any one or more surrogates is outside the following acceptance criteria:

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<u>Surrogate</u>	<u>Acceptance Criteria % Low-Level</u>
Naphthalene-d8	14-108
Fluorene-d10	41-162
Chrysene-d12	10-118

The following corrective action will be taken when required as stated above:

- a) Check calculations to assure there are no errors;
- b) Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
- c) Reanalyze the sample or extract if the steps in part a) or b) fail to reveal a problem. If reanalysis of the extracts yields surrogate spike recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported.
- d) If a), b) or c) do not correct the problem, the data for that sample will be reported, but documented as being outside the acceptance criteria limits.

11.5 Matrix Spike Analysis

The laboratory will spike and analyze 5% matrix spike samples. RMAL will spike seven representative compounds into water. These compounds and the spiking levels are listed below:

Naphthalene	20 ng/L
Fluorene	20
Chrysene	20
Indene	20
Quinoline	20
Benz(e)pyrene	20
2-methyl naphthalene	20

The initial matrix spike criteria for data validity are as follows:

- o The average of the percent recoveries for all seven compounds must fall between 20 and 150 percent.
- o Only one compound can be below its required minimum percent recovery. These minimum percent recoveries are:

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- 1) 10% for chrysene and benz(e)pyrene
- 2) 20% for all other compounds.

Criteria for data validity for each individual matrix spike compound will be developed as data is collected and will be updated on a quarterly basis.

If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, then the reanalysis data will be used. If not, the data for the sample will be reported but qualified as being outside the acceptance criteria of the method. Both the original and reanalysis data will be reported.

Table 4 contains percent recovery results for the target compounds spiked into reagent water at levels near the method detection limits (i.e. 2.5 to 5.0 ng/L).

11.6 Duplicates

The laboratory will analyze 10% duplicate samples. Percent difference between duplicates will be calculated for each detected compound.

12.0 Data Deliverables

Data is presented in the format described in Exhibit B of Organic SOW 7/87 for the Contract Lab Program. The various items in the data package are listed below:

- 1) Sample Traffic Reports or Chain-of-Custody
- 2) Sample Data Summary Package Including:
 - Case narrative
 - Tabulated target compound results by fraction
 - Surrogate spike analysis results by fraction
 - Matrix spike/matrix spike duplicate results by fraction
 - Blank data by fraction
- 3) Sample Data Package including:
 - Case narrative
 - Traffic reports
 - Volatiles Data
 - Semivolatiles Data
 - Pesticide Data

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The volatiles, semivolatiles and pesticides data packages will include a QC summary, the raw sample data, standards data and raw QC data.

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TABLE 1

COMPOUNDS AND MS QUANTITATION MASS IONS

<u>Compound</u>	<u>Quantitation Mass Ion</u>	<u>Confirmation Ion (% Abundance)</u>	<u>Internal Standard Reference</u>
<u>Polynuclear Aromatic Hydrocarbons (PAH)</u>			
Naphthalene	128	102 (7)*	1
Acenaphthylene	152	151 (17)*	1
Acenaphthene	154	153 (93)*	1
Fluorene	166	165 (90)*	1
Phenanthrene	178	176 (19)*	2
Anthracene	178	176 (19)*	2
Fluoranthene	202	200 (17)*	2
Pyrene	202	200 (18)*	2
Benzo(a)anthracene	228	226 (22)*	3
Chrysene	228	226 (26)*	3
Benzofluoranthenes	252	250 (22)*	3
Benzo(a)pyrene	252	250 (26)*	3
Indeno(1,2,3,cd)pyrene	276	274 (21)*	3
Dibenz(a,h)anthracene	278	279 (20)*	3
Benzo(g,h,i)perylene	276	274 (25)*	3

Internal Standards

1) Acenaphthene-d10	164	--
2) Phenanthrene-d10	188	--
3) Benzo(a)pyrene-d12	264	--

Surrogates

1) Naphthalene-d8	136	1
2) Fluorene-d10	176	1
3) Chrysene-d12	240	3

*The % abundance for the confirmation ion is a typical value obtained during the method detection limit study. Although these ratios will vary, the relative intensities of confirmation ions must agree within plus or minus 20% between the calibration standard for any given day and the samples run on that day.

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TABLE 1 (Continued)

<u>Compound</u>	<u>Quantitation Mass Ion</u>	<u>Confirmation Ion (% Abundance)</u>	<u>Internal Standard Reference</u>
<u>Heterocycles and Other PAH</u>			
Indene	116	115 (108)*	1
Indole	117	90 (52)*	1
2,3-dihydroindene	117	118 (57)*	1
2,3-benzofuran	118	90 (31)*	1
Quinoline	129	102 (20)*	1
Benzo(b)thiophene	134	89 (8)*	1
2-methylnaphthalene	141	115 (31)*	1
1-methylnaphthalene	141	115 (28)*	1
Biphenyl	154	153 (35)*	1
Carbazole	167	166 (28)*	2
Dibenzofuran	168	139 (40)*	1
Acridine	179	178 (26)*	2
Dibenzothiophene	184	139 (19)*	2
Perylene	252	250 (24)*	3
Benzo(e)pyrene	252	250 (35)*	3

Internal Standards

1) Acenaphthene-d10	164	--
2) Phenanthrene-d10	188	--
3) Benzo(a)pyrene-d12	264	--

Surrogates

1) Naphthalene-d8	136	1
2) Fluorene-d10	176	2
3) Chrysene-d12	240	3

*The % abundance for the confirmation ion is a typical value obtained during the method detection limit study. Although these ratios will vary, the relative intensities of confirmation ions must agree within plus or minus 20% between the calibration standard for any given day and the samples run on that day.

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TABLE 2

RELATIVE RETENTION TIMES AND CONFIDENCE FOR THE COMPOUNDS
ASSOCIATED WITH THE LOW LEVEL PAH AND HETEROCYCLE METHODOLOGY

	<u>Absolute Retention Time (minutes)</u>	<u>Avg. RRT</u>	<u>SD</u>	<u>% RSD</u>	<u>95% Confidence Limits</u>
Benzofuran	8:03	0.550	0.015	2.807	0.520-0.580
Dihydroindene	8:45	0.590	0.016	2.765	0.558-0.622
Indene	8:54	0.598	0.016	2.699	0.566-0.630
Naphthalene-d8(Surr.)	11:14	0.733	0.017	2.289	0.699-0.767
Naphthalene	11:16	0.735	0.017	2.289	0.701-0.769
Benzo(b)thiophene	11:25	0.743	0.017	2.258	0.709-0.777
Quinoline	12:06	0.783	0.017	2.140	0.749-0.817
Indole	12:55	0.824	0.018	2.167	0.788-0.860
2-methylnaphthalene	12:59	0.832	0.017	2.084	0.798-0.866
1-methylnaphthalene	13:15	0.848	0.017	2.055	0.814-0.882
Biphenyl	14:12	0.901	0.017	1.921	0.867-0.935
Acenaphthylene	15:15	0.962	0.018	1.822	0.927-0.988
Acenaphthene	15:44	0.988	0.018	1.849	0.952-1.024
Dibenzofuran	16:09	1.011	0.018	1.791	0.975-1.047
Fluorene-d10(Surr.)	16:57	0.872	0.015	1.735	0.842-0.902
Fluorene	17:01	0.875	0.015	1.745	0.845-0.905
Dibenzothiophene	19:08	0.974	0.016	1.617	0.942-1.006
Phenanthrene	19:28	0.988	0.016	1.589	0.956-1.020
Anthracene	19:34	0.994	0.016	1.597	0.962-1.026
Acridine	19:42	0.999	0.016	1.572	0.967-1.031
Carbazole	20:02	1.013	0.015	1.487	0.983-1.043
Fluoranthene	22:32	1.130	0.017	1.461	1.096-1.164
Pyrene	23:07	1.157	0.017	1.443	1.123-1.191
Benz(a)anthracene	26:16	0.873	0.012	1.325	0.849-0.897
Chrysene-d12 (Surr.)	26:18	0.874	0.012	1.320	0.850-0.898
Chrysene	26:22	0.876	0.012	1.320	0.852-0.900
Benzofluoranthenes	29:00	0.960	0.014	1.501	0.932-0.988
Benzo(e)pyrene	29:34	0.984	0.016	1.590	0.952-1.016
Benzo(a)pyrene	29:44	0.988	0.016	1.615	0.956-1.020
Perylene	29:55	0.996	0.016	1.644	0.964-1.028
Indeno(1,2,3 cd)pyrene	32:31	1.114	0.025	2.276	1.064-1.164
Dibenz(ah)anthracene	32:36	1.113	0.031	2.743	1.051-1.175
Benzo(ghi)perylene	33:17	1.149	0.028	2.422	1.093-1.205

Title: DETERMINATION OF LOW LEVEL (PART PER TRILLION)
PAH AND HETEROCYCLES IN WATER

TABLE 3
SELECTED ION MONITORING (SIM) SEQUENCES FOR PAH
AND HETEROCYCLES

<u>Sequence #</u>	<u>M/Z Scanned</u>	<u>Scan # Range</u>	<u>Start Time (min)</u>
1	90, 115, 116, 117, 118	0 - 399	0:00
2	90, 115, 116, 117, 118	400 - 849	4:40
3	89, 102, 128, 129, 134, 136	850 - 1064	9:55
4	90, 115, 117, 141	1065 - 1169	12:26
5	151, 152, 153, 154, 164	1170 - 1354	13:39
6	139, 165, 166, 168, 176	1355 - 1524	15:49
7	139, 176, 178, 179, 184, 188	1525 - 1684	17:48
8	166, 167	1685 - 1799	19:40
9	200, 202, 226, 228, 240	1800 - 2299	21:00
10	241, 250, 252, 256	2300 - 2479	26:50
11	250, 252, 264, 268	2480 - 2649	28:56
12	274, 276, 278, 279, 302, 303	2650 - 3500	30:55

STANDARD OPERATING PROCEDURE

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**Title: DETERMINATION OF LOW LEVEL (PART PER TRILLION)
PAH AND HETEROCYCLES IN WATER**

**TABLE 4
LOW LEVEL PNA SPIKE RESULTS**

<u>Compound</u>	Sample <u>#1</u>	Sample <u>#2</u>	Sample <u>#3</u>	Percent Recovery		
				Sample <u>#1</u>	Sample <u>#2</u>	Sample <u>#3</u>
2,3-Benzofuran	2.5	2.3	2.6	101	90	105
2,3-Dihydroindene	2.9	2.6	3.0	115	105	119
1H-Indene	2.2	2.2	2.7	89	89	109
Naphthalene	5.9*	N/A	N/A	118	N/A	N/A
Benzo(B)thiophene	1.7	1.9	1.8	68	74	73
Quinoline	2.5	2.2	2.0	101	89	81
1H-Indole	1.5	2.2	2.7	60	90	107
2-Methylnaphthalene	2.3	2.5	2.7	92	99	107
1-Methylnaphthalene	2.2	2.3	2.5	88	91	99
Biphenyl	4.2*	N/A	N/A	83	N/A	N/A
Acenaphthylene	2.0	1.9	2.1	79	76	83
Acenaphthene	2.3	2.5	2.4	91	100	95
Dibenzofuran	1.6	1.6	1.5	64	62	61
Fluorene	2.0	2.1	2.3	82	82	93
Dibenzothiophene	1.7	1.7	1.8	67	69	72
Phenanthrene	1.9	2.1	2.5	75	82	100
Anthracene	1.6	1.7	2.4	65	67	96
Acridine	0.9	1.0	1.2	37	41	47
Carbazole	1.3	1.3	1.3	50	53	53
Fluoranthene	1.9	2.5	2.4	75	101	96
Pyrene	1.9	3.2	2.4	77	128	97
Benzo(A)anthracene	2.5	2.5	2.8	100	101	113
Chrysene	2.4	2.2	2.6	95	90	103
Benzo(B)fluoranthrene	1.8	1.8	2.3	71	72	93
Benzo(K)fluoranthrene	2.2	2.2	2.3	89	89	90
7,12-Dimethylbenzanthracene	3.3	3.6	3.3	132	142	133
Benzo(E)pyrene	1.9	1.8	2.0	75	73	80
Benzo(A)pyrene	1.9	2.0	2.4	78	79	96
Perylene	2.2	2.1	2.6	89	82	102
3-Methylcholanthrene	2.2	2.3	2.1	88	90	82
Indeno(1,2,3-CD)pyrene	2.0	1.9	2.1	82	75	84
Dibenz(A,C)anthracene	1.7	1.8	2.0	68	72	80
Dibenz(A,H)anthracene	1.7	1.8	2.0	67	72	80
Benzo(G,H,I)perylene	2.2	2.1	2.3	90	85	91

Note: All compounds spiked at 2.5 ng/L.

*Data for Naphthalene and Biphenyl were obtained from previous study.

Spike levels = 5.0 ng/L.

N/A = Not applicable.

Section 4
Standard Operating Procedure
part-per-billion PNAs

EXHIBIT D

**ANALYTICAL METHODS
FOR SEMIVOLATILES**

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SECTION I

INTRODUCTION

The analytical methods that follow are designed to analyze water, soil and sediment from hazardous waste sites for the organic compounds on the Target Compound List (TCL) (See Exhibit C). The methods are based on EPA Method 625 (Base/Neutrals and Acids).

The methods are divided into the following sections: sample preparation, screening, and analysis. Sample preparation covers sample extraction and cleanup techniques. As described in the screening section, a portion of the extracts may be screened on a gas chromatograph with appropriate detectors to determine the concentration level of organics. The analysis section contains the GC/MS analytical methods for organics.

1. Method for the Determination of Extractable Semivolatiles (Base/Neutral and Acid) Organic Compounds.

1.1 Scope and Application

This method covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. These TCL compounds and the contract required quantitation limits are listed in Exhibit C.

Problems have been associated with the following compounds covered by this method. Dichlorobenzidine and 4-chloroaniline can be subject to oxidative losses during solvent concentration. This is especially true in the soil/sediment method when concentrating the methylene chloride/acetone extraction solvent. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet forming diphenylamine and, consequently, cannot be separated from diphenylamine native to the sample.

- 1.2 The method involves solvent extraction of the matrix sample characterization to determine the appropriate analytical protocol to be used, and GC/MS analysis to determine semivolatile (BNA) organic compounds present in the sample.**

SECTION II

SAMPLE PREPARATION AND STORAGE

PART A - SAMPLE STORAGE AND HOLDING TIMES**1. Procedures for Sample Storage**

- 1.1 The samples must be protected from light and refrigerated at 4°C (+2°C) from the time of receipt until extraction and analysis.
- 1.2 After analysis, extracts and unused sample volume must be protected from light and refrigerated at 4°C (+2°C) for the periods specified in the contract schedule.

2. Contract Required Holding Times

- 2.1 If separatory funnel or sonication procedures are employed for extractions for semivolatile analyses, extraction of water samples shall be completed within 5 days of VTSR (Validated Time of Sample Receipt), and extraction of soil/sediment samples shall be completed within 10 days of VTSR. If continuous liquid-liquid extraction procedures are employed, extraction of water samples shall be started within 5 days of VTSR.

Extracts of either water or soil/sediment samples must be analyzed within 40 days of VTSR.

PART B - SAMPLE PREPARATION FOR EXTRACTABLE SEMIVOLATILES (BNA) IN WATER**1. Summary of Method**

A measured volume of sample, approximately one liter, is serially extracted with methylene chloride at a pH greater than 11 and again at pH less than 2, using a separatory funnel or a continuous extractor. The methylene chloride extracts are dried and concentrated separately to a volume of 1 mL.

2. Interferences

2.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware, that lead to discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source.

3. Apparatus and Materials

3.1 Glassware (Brand names and catalog numbers are included for illustration purposes only).

3.1.1 Separatory funnel - 2,000 mL, with teflon stopcock.

3.1.2 Drying column - 19 mm ID chromatographic column with coarse frit. (Substitution of a small pad of Pyrex glass wool for the frit will prevent cross contamination of sample extracts.)

3.1.3 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

3.1.4 Evaporative flask - Kuderna-Danish, 500 mL (Kontes K-570001 0500 or equivalent). Attach to concentrator tube with springs.

3.1.5 Snyder column - Kuderna-Danish, Three-ball macro (Kontes K-503000 0121 or equivalent).

3.1.6 Snyder column - Kuderna-Danish, Two-ball micro (Kontes K569001 0219 or equivalent).

3.1.7 Vials - Amber glass, 2 mL capacity with Teflon-lined screw cap.

- 3.1.8 Continuous liquid-liquid extractors - Equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf Extractor-Ace Glass Company, Vineland, NJ P/N 6841-10 or equivalent.)
- 3.2 Silicon carbide boiling chips - approximately 10/40 mesh. Heat to 400 °C for 30 minutes or Soxhlet extract with methylene chloride.
- 3.3 Water bath - Heated, with concentric ring cover, capable of temperature control ($\pm 2^{\circ}\text{C}$). The bath should be used in a hood.
- 3.4 Balance - Analytical, capable of accurately weighing ± 0.0001 g.
- 3.5 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C. The N-Evap by Organomation Associates, Inc. South Berlin, MA (or equivalent) is suitable.

4. Reagents

- 4.1 Reagent water - Reagent water is defined as a water in which an interferent is not observed at or above the CRQL of each parameter of interest.
- 4.2 Sodium hydroxide solution (10N) - Dissolve 40 g NaOH in reagent water and dilute to 100 mL.
- 4.3 Sodium thiosulfate - (ACS) Granular.
- 4.4 Sulfuric acid solution (1+1) - Slowly add 50 mL of H_2SO_4 (sp gr. 1.84) to 50 mL of reagent water.
- 4.5 Acetone, methanol, methylene chloride - Pesticide quality or equivalent.
- 4.6 Sodium sulfate - (ACS) Powdered, anhydrous. Purify by heating at 400°C for four hours in a shallow tray, cool in a desiccator, and store in a glass bottle. Baker anhydrous powder, catalog #73898 or equivalent.
- 4.7 Surrogate standard spiking solution.
 - 4.7.1 Surrogate standards are added to all samples and calibration solutions; the compounds specified for this purpose are phenol- d_6 ; 2,4,6 tribromophenol; 2 fluorophenol; nitrobenzene- d_5 ; terphenyl- d_{14} and 2-fluorobiphenyl. Two additional surrogates, one base/neutral and one acid, may be added.
 - 4.7.2 Prepare a surrogate standard spiking solution that contains the base/neutral compounds at a concentration of 100 ug/mL, and the acid compounds at 200 ug/mL. Store the spiking solutions at 4°C ($\pm 2^{\circ}\text{C}$) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.
- 4.8 BNA Matrix standard spiking solution. The matrix spike solution consists of:

Base/Neutrals

1,2,4-trichlorobenzene
 acenaphthene
 2,4-dinitrotoluene
 pyrene
 N-nitroso-di-n-propylamine
 1,4-dichlorobenzene

Acids

pentachlorophenol
 phenol
 2-chlorophenol
 4-chloro-3-methylphenol
 4-nitrophenol

Prepare a spiking solution that contains each of the base/neutral compounds above at 100 ug/1.0 mL in methanol and the acid compounds at 200 ug/1.0 mL in methanol. Analyze duplicate aliquots of a sample spiked with BNA matrix spiking solution.

5. Sample Extraction - Separatory Funnel

- 5.1 Samples may be extracted using separatory funnel techniques. If emulsions prevent acceptable solvent recovery with separatory funnel extraction, continuous extraction (paragraph 6.) may be used. The separatory funnel extraction scheme described below assumes a sample volume of 1-liter.
- 5.2 Using a 1-liter graduated cylinder, measure out a 1-liter sample aliquot and place it into a 2-liter separatory funnel. Pipet 1.0 mL surrogate standard spiking solution into the separatory funnel and mix well. Check the pH of the sample with wide range pH paper and adjust to pH > 11 with 10N sodium hydroxide. Add 1.0 mL of BNA matrix spiking solution to each of two 1-liter portions from the sample selected for spiking.
- 5.3 Add 60 mL methylene chloride to the separatory funnel and extract the sample by shaking the funnel for two minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include: stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.
- Collect the methylene chloride extract in a 250-mL Erlenmeyer flask. If the emulsion cannot be broken (recovery of less than 80% of the methylene chloride, corrected for the water solubility of methylene chloride), transfer the sample, solvent and emulsion into the extraction chamber of a continuous extractor. Proceed as described in paragraph 6.3.
- 5.4 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner. Label the combined extract as the base/neutral fraction.

- 5.5 Adjust the pH of the aqueous phase to less than 2 using sulfuric acid (1 + 1). Serially extract three times with 60-mL aliquots of methylene chloride, as per paragraph 5.3. Collect and combine the extracts in a 250-mL Erlenmeyer flask and label the combined extract as the acid fraction.
- 5.6 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D, if equivalency is demonstrated for all extractable organics listed in Exhibit C.
- 5.7 Transfer the individual base/neutral and acid fractions by pouring extracts through separate drying columns containing about 10 cm of anhydrous granular sodium sulfate, and collect the extracts in the separate K-D concentrators. Rinse the Erlenmeyer flasks and columns with 20 to 30 mL of methylene chloride to complete the quantitative transfer.
- 5.8 Add one or two clean boiling chips and attach a three-ball Snyder column to the evaporative flask. Pre-wet the Snyder column by adding about 1 mL methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80° to 90°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of methylene chloride. A 5-mL syringe is recommended for this operation.
- 5.9 Micro Snyder column technique - Add another one or two clean boiling chips to the concentrator tube and attach a two-ball micro Snyder column. Pre-wet the Snyder column by adding about 0.5 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80° to 90°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches about 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column and rinse its flask and its lower joint into the concentrator tube with 0.2 mL of methylene chloride. Adjust the final volume to 1.0 mL with methylene chloride. If GC/MS analysis will not be performed immediately, stopper the concentrator tube and store refrigerated. If the extracts will be stored longer than two days, they should be transferred to individual Teflon-sealed screw cap bottles and labeled base/neutral or acid fraction, as appropriate.

5.10 Nitrogen blowdown technique (taken from ASTM Method D3086)

The following method may be used for final concentration, instead of the procedure outlined in paragraph 5.9. Place the concentrator tube in a warm water bath (35°C) and evaporate the solvent volume to just below 1 mL using a gentle stream of clean, dry nitrogen filtered through a column of activated carbon). Caution: New plastic tubing must not be used between the carbon trap and the sample, as it may introduce interferences. The internal wall of the tube must be rinsed down several times with methylene chloride during the operation and the final volume brought to 1 mL with methylene chloride. During evaporation, the tube solvent level must be kept below the water level of the bath. The extract must never be allowed to become dry.

6. Sample Extraction - Continuous Liquid-Liquid Extractor

- 6.1 Check the pH of the sample with wide-range pH paper and adjust to pH 11 with 10 N sodium hydroxide. Transfer a 1-liter sample aliquot to the continuous extractor; using a pipet, add 1 mL of surrogate standard spiking solution and mix well.
- 6.2 Add 500 mL of methylene chloride to the distilling flask. Add sufficient reagent water to ensure proper operation and extract for 18 hours. Allow to cool, then detach the boiling flask and dry. Concentrate the extract as in paragraphs 5.6 through 5.8. Hold the concentrated extract for combining with the acid extract (see paragraph 6.4).
- 6.3 Add 500 mL of methylene chloride to a clean distilling flask and attach it to the continuous extractor. Carefully adjust the pH of the aqueous phase to less than 2 using sulfuric acid (1 + 1). Extract for 18 hours. Dry and concentrate the extract as described in paragraphs 5.6 through 5.8. Hold the concentrated extract and label as the acid extract.
 - 6.3.1 If the base/neutral and/or acid extracts cannot be concentrated to a final volume of 1 mL, dilute the more concentrated extract to the final volume of the least concentrated extract.

7. The samples extracts are ready for GC/MS analysis. Proceed to Section IV, GC/MS Analysis of Semivolatiles. If high concentrations are suspected (e.g., highly colored extracts) the optional GC/FID screen in Section III is recommended.

PART C - PROTOCOLS FOR SOIL/SEDIMENT

It is mandatory that all soil/sediment samples be characterized as to concentration level so that the appropriate analytical protocol is chosen to ensure proper quantitation limits for the sample. Note that the terms "low level" and "medium level" are not used here as a judgement of degree of contamination but rather as a description of the concentration ranges that are encompassed by the "low" and "medium" level procedures.

The laboratory is at liberty to determine the method of characterization. The following two screening methods may be used for soil/sediment sample characterization:

- o Screen an aliquot from the "low level" 30 g extract or an aliquot from the "medium level" 1 g extract.
- o Screen using either GC/FID or GC/MS as the screening instrument.

The concentration ranges covered by these two procedures may be considered to be approximately 330 ug/kg - 20,000 ug/kg for the low level analysis and >20,000 ug/kg for medium level analysis for BNA extractables. For soils only, the extract for pesticide/PCB analysis may be prepared from an aliquot of the extract for semivolatiles, or in a separate extraction procedure. If it is prepared from the semivolatile extract, refer to Exhibit D PEST for the procedures for extraction of pesticides/PCBs.

Screen from the Medium Level Method

Take 5.0 mL from the 10.0 mL total extract and concentrate to 1.0 mL and screen. If the sample concentration is >20,000 ug/kg proceed with GC/MS analysis of the organics. If the sample concentration is <20,000 ug/kg discard the medium level extract and follow the low level method.

Screen from Low Level Method

Take 5.0 mL from the 300 mL (approximate) total extract from the 30 g sample and concentrate to 1.0 mL and screen. If the concentration is >20,000 ug/kg in the original sample, discard the 30 g extract and follow the medium level methods for organics, using medium level surrogates. If the sample concentration is <20,000 ug/kg, proceed with concentration and the remainder of the low level method.

1. Medium Level Preparation for Screening and Analysis of Semivolatiles (BNA)**1.1 Scope and Application**

This procedure is designed for the preparation of sediment/soil samples which may contain organic chemicals at a level greater than 20,000 ug/kg.

- 1.1.1 The extracts and sample aliquots prepared using this method are screened by GC/MS or FID, using capillary columns for base/neutral and acid priority pollutants, and related organic chemicals. The results of these screens will determine whether sufficient quantities of pollutants are present to warrant analysis by low or medium protocol.

- 1.1.2 If the screenings indicate no detectable pollutants at the lower limits of quantitation, the sample should be prepared by the low level protocol in Section II, Part C, paragraph 2.

1.2 Summary of Method

- 1.2.1 Approximately 1 g portions of sediment/soil are transferred to vials and extracted with methylene chloride. The methylene chloride extract is screened for extractable organics by GC/FID or GC/MS.
- 1.2.2 If organic compounds are detected by the screen, the methylene chloride extract is analyzed by GC/MS for extractable organics.
- 1.2.3 If no organic compounds are detected by the medium level screen, then a low level sample preparation is required.

1.3 Interferences

- 1.3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source.

1.4 Limitations

- 1.4.1 The procedure is designed to allow quantitation limits for screening purposes as low as 20,000 ug/kg for extractable organics. For analysis purposes, the quantitation limits are 20,000 ug/kg for extractable organics. If peaks are present based on the GC/FID screen, the sample is determined to require a medium level analysis by GC/MS. Some samples may contain high concentrations of chemicals that interfere with the analysis of other components at lower levels; the quantitation limits in those cases may be significantly higher.
- 1.4.2 These extraction and preparation procedures were developed for rapid and safe handling of high concentration hazardous waste samples. The design of the methods thus does not stress efficient recoveries or low limits of quantitation of all components. Rather, the procedures were designed to screen at moderate recovery and sufficient sensitivity, a broad spectrum of organic chemicals. The results of the analyses thus may reflect only a minimum of the amount actually present in some samples.

1.5 Reagents

1.5.1 Sodium Sulfate - anhydrous powdered reagent grade, heated at 400°C for four hours, cooled in a desiccator, and stored in a glass bottle Baker anhydrous powder, catalog # 73898 or equivalent.

1.5.2 Methylene chloride. Pesticide residue analysis grade or equivalent.

1.5.3 Methanol. Pesticide residue analysis grade or equivalent.

1.5.4 Acetone. Pesticide residue analysis grade or equivalent.

1.5.5 Base/Neutral and Acid Surrogate Standard Spiking Solution

The compounds specified are phenol-d₆, 2,4,6-tribromophenol, 2-fluorophenol, nitrobenzene-d₅, terphenyl-d₁₄ and 2-fluorobiphenyl. Prepare a solution containing these compounds for base/neutral surrogates at a concentration of 100 ug/1.0 mL, and for acid surrogate standards at a concentration of 200 ug/1.0 mL in methanol. Store the spiking solutions at 4°C (+2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner, if comparison with quality control check samples indicates a problem.

1.5.6 Base/Neutral and Acid Matrix Standard Spiking solution.

Prepare a spiking solution in methanol that contains the following compounds at a concentration of 100 ug/1.0 mL for base/neutrals and 200 ug/1.0 mL for acids. Store the spiking solutions at 4°C (+2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner, if comparison with quality control check samples indicates a problem.

Base Neutrals

1,2,4-trichlorobenzene
acenaphthene
2,4-dinitrotoluene
pyrene
N-nitroso-di-n-propylamine
1,4-dichlorobenzene

Acids

pentachlorophenol
phenol
2-chlorophenol
4-chloro-3-methylphenol
4-nitrophenol

1.6 Equipment

1.6.1 Glass scintillation vials, at least 20 mL, with screw cap and teflon or aluminum foil liner.

- 1.6.2 Spatula. Stainless steel or Teflon.
- 1.6.3 Balance capable of weighing 100 g to ± 0.01 g.
- 1.6.4 Vials and caps, 2 mL for GC auto sampler.
- 1.6.5 Disposable pipets, Pasteur; glass wool rinsed with methylene chloride.
- 1.6.6 15-mL concentrator tubes.
- 1.6.7 Ultrasonic cell disruptor, Heat Systems Ultrasonics, Inc., Model W-385 SONICATOR (475 Watt with pulsing capability, No. 200 1/2 inch tapped disruptor horn plus No. 207 3/4 inch tapped disruptor horn, and No. 419 1/8 inch standard tapered MICROTIP probe), or equivalent device with a minimum of 375 Watt output capability. NOTE: In order to ensure that sufficient energy is transferred to the sample during extraction, the MICROTIP probe must be replaced if the tip begins to erode. Erosion of the tip is evidenced by a rough surface.
- 1.6.8 Sonabox acoustic enclosure - recommended with above disruptors for decreasing cavitation sound.
- 1.6.9 Test tube rack.
- 1.6.10 Oven, drying.
- 1.6.11 Desiccator.
- 1.6.12 Crucibles, porcelain.

1.7 Medium Level Sample Preparation.

- 1.7.1 Transfer the sample container into a fume hood. Open the sample vial. Decant and discard any water layer and then mix the sample. Transfer approximately 1 g (record weight to the nearest 0.1 g) of sample to a 20-mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample taken. Cap the vial before proceeding with the next sample to avoid any cross-contamination.
 - 1.7.1.1 Transfer 50 g of soil/sediment to 100 mL beaker. Add 50 mL of water and stir for 1 hour. Determine pH of sample with glass electrode and pH meter while stirring. Report pH value on appropriate data sheets. If the pH of the soil is greater than 11 or less than 5, contact the Deputy Project Officer cited in the contract for instructions on how to handle the sample. Document the instructions in the Case Narrative. Discard this portion of sample.

II.C

- 1.7.2 Immediately after weighing the sample for extraction, weigh 5-10 g of the sediment into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100 = \% \text{ moisture}$$

- 1.7.3 Add 2.0 g of anhydrous powdered sodium sulfate to sample in the 20 mL vial from paragraph 1.7.1 and mix well.

- 1.7.4 Surrogate Standards are added to all samples, spikes, and blanks. Add 1.0 mL of surrogate spiking solution to sample mixture.
- 1.7.5 Add 1.0 mL of matrix standard spiking solution to each of two 1 g portions from the sample chosen for spiking.
- 1.7.6 Immediately add 9.0 mL of methylene chloride to the sample and disrupt the sample with the 1/8 inch tapered MICROTIP ultrasonic probe for 2 minutes at output control setting 5, in continuous mode. (If using a sonicator other than Models W-375 or W-385, contact the Project Officer for appropriate output settings). Before extraction, make certain that the sodium sulfate is free flowing and not a consolidated mass. As required, break up large lumps with a clean spatula, or very carefully with the tip of the unenergized probe.
 - 1.7.6.1 Add only 8.0 mL of methylene chloride to the matrix spike samples to achieve a final volume of 10 mL.
- 1.7.7 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extract through the glass wool and collect 5.0 mL in a concentrator tube.
- 1.7.8 Concentrate the extract to 1.0 mL by the nitrogen blowdown technique described in paragraph 2.7.3.
- 1.7.9 Transfer the concentrate to an autosampler vial for GC/FID or GC/MS capillary column screening. If the concentrate is screened, the quantitation limits should be approximately 20,000 ug/kg.
- 1.7.10 Proceed to Section III, paragraph 1.

2. Low Level Preparation for Screening and Analysis of Semivolatiles (BNA)

2.1 Summary of Method

A 30 gram portion of sediment is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone using an ultrasonic probe. If the optional low level screen is used, a portion of this dilute extract is concentrated fivefold and is screened by GC/FID or GC/MS. If peaks are present at greater than 20,000 ug/kg, discard the extract and prepare the sample by the medium level method. If no peaks are present at greater than 20,000 ug/kg, the extract is concentrated. An optional gel permeation column cleanup may be used before analysis.

2.2 Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles.

All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source.

2.3 Apparatus and Materials

2.3.1 Apparatus for determining percent moisture

2.3.1.1 Oven, drying

2.3.1.2 Desiccator

2.3.1.3 Crucibles, porcelain

2.3.2 Disposable Pasteur glass pipets, 1 mL

2.3.3 Ultrasonic cell disruptor, Heat Systems - Ultrasonics, Inc. Model 385 SONICATOR (475 Watt with pulsing capability, No. 305 3/4 inch tapped high gain "Q" disruptor horn or No. 208 3/4 inch standard solid disruptor horn), or equivalent device with a minimum of 375 Watt output capability. NOTE: In order to ensure that sufficient energy is transferred to the sample during extraction, the horn must be replaced if the tip begins to erode. Erosion of the tip is evidenced by a rough surface.

2.3.3.1 Sonobox acoustic enclosure - recommended with above disruptors for decreasing cavitation sound.

2.3.4 Beakers, 400 mL

2.3.5 Vacuum filtration apparatus

2.3.5.1 Buchner funnel.

2.3.5.2 Filter paper, Whatman No. 41 or equivalent.

2.3.6 Kuderna-Danish (K-D) apparatus.

2.3.6.1 Concentrator tube - 10 mL, graduated (Kontes K-570040-1025 or equivalent).

2.3.6.2 Evaporative flask - 500 mL (Kontes K-570001-0500 or equivalent).

2.3.6.3 Snyder column - three-ball macro (Kontes K-50300C-0121 or equivalent).

2.3.6.4 Snyder column - two-ball micro (Kontes K-569001-0219) or equivalent).

2.3.7 Silicon carbide boiling chips - approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.

- 2.3.8 Water bath - heated, with concentric ring cover, capable of temperature control ($\pm 2^{\circ}\text{C}$). The bath should be used in a hood.
- 2.3.9 Balance, capable of accurately weighing ± 0.01 g.
- 2.3.10 Vials and caps, 2 mL for GC auto sampler.
- 2.3.11 Balance - Analytical, capable of accurately weighing ± 0.0001 g.
- 2.3.12 Nitrogen evaporation device equipped with a water bath that can be maintained at $35-40^{\circ}\text{C}$. The N-Evap by Organomation Associates, Inc. South Berlin, MA (or equivalent) is suitable.
- 2.3.13 Gel permeation chromatography (GPC) cleanup device. NOTE: GPC cleanup is highly recommended for all extracts for low level soils.

2.3.13.1 Automated system

2.3.13.1.1 Gel permeation chromatograph Analytical Biochemical Labs, Inc. GPC Autoprep 1002 or equivalent including:

2.3.13.1.2 25 mm ID X 600 - 700 mm glass column packed with 70 g of Bio-Beads SX-3.

2.3.13.1.3 Syringe, 10 mL with Luer-Lok fitting.

2.3.13.1.4 Syringe filter holder and filters - stainless steel and TFE, Gelman 4310 or equivalent.

2.3.13.2 Manual system assembled from parts.*

2.3.13.2.1 25 mm ID X 600 - 700 mm heavy wall glass column packed with 70 g of BIO-Beads SX-3.

2.3.13.2.2 Pump: Altex Scientific, Model No. 1001A, semipreparative, solvent metering system. Pump capacity = 28 mL/min.

2.3.13.2.3 Detector: Altex Scientific, Model No. 153, with 254 nm UV source and 8-ul semi-preparative flowcells (2-mm pathlengths)

2.3.13.2.4 Microprocessor/controller: Altex Scientific, Model No. 420, Microprocessor System Controller, with extended memory.

*Wise, R.H., Bishop, D.F., Williams, R.T. & Austern, B.M. "Gel Permeation Chromatography in the GC/MS Analysis of Organics in Sludges" U.S. EPA, Municipal Environmental Research Laboratory - Cincinnati, Ohio 45268

- 2.3.13.2.5 Injector: Altex Scientific, catalog No. 201-56, sample injection valve, Tefzel, with 10 mL sample loop.
- 2.3.13.2.6 Recorder: Linear Instruments, Model No. 385, 10-inch recorder.
- 2.3.13.2.7 Effluent Switching Valve: Teflon slider valve, 3-way with 0.060" ports.
- 2.3.13.2.8 Supplemental Pressure Gauge with connecting Tee: U.S. Gauge, 0-200 psi, stainless steel. Installed as a "downstream" monitoring device between column and detector.

Flow rate was typically 5 mL/min. of methylene chloride. Recorder chart speed was 0.50 cm/min.

2.3.14 Pyrex glass wool.

2.3.15 Pasteur pipets, disposable.

2.4 Reagents

- 2.4.1 Sodium Sulfate - anhydrous powdered reagent grade, heated at 400°C for four hours, cooled in a desiccator, and stored in a glass bottle. Baker anhydrous powder, catalog #73898 or equivalent.
- 2.4.2 Methylene chloride, methanol, acetone, isooctane, 2-propanol and benzene pesticide quality or equivalent.
- 2.4.3 Reagent water - Reagent water is defined as a water in which an interferent is not observed at or above the CRQL of each parameter of interest.
- 2.4.4 GPC calibration solutions:
 - 2.4.4.1 Corn oil - 200 mg/mL in methylene chloride.
 - 2.4.4.2 Bis(2-ethylhexylphthalate) and pentachlorophenol - 4.0 mg/mL in methylene chloride.
- 2.4.5 Sodium Sulfite, reagent grade.
- 2.4.6 Surrogate standard spiking solution.
 - 2.4.6.1 Base/neutral and acid surrogate solution.

2.4.6.1.1 Surrogate standards are added to all samples, blanks, matrix spikes, matrix spike duplicates, and calibration solutions; the compounds specified for this purpose are phenol-d₆, 2,4,6-tribromophenol, 2-fluorophenol, nitrobenzene-d₅, terphenyl-d₁₄, and 2-fluorobiphenyl. Two additional surrogates, one base/neutral and one acid may be added.

2.4.6.1.2 Prepare a surrogate standard spiking solution at a concentration of 100 ug/1.0 mL for base/ neutral and 200 ug/1.0 mL for acids in methanol. Store the spiking solutions at 4°C (+2°C) in Teflon-sealed containers. The solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicate a problem.

2.4.7 Matrix standard spiking solutions.

2.4.7.1 Base/neutral and acid matrix spiking solution consists of:

<u>Base/Neutrals</u> (100 ug/1.0 mL)	<u>Acids</u> (200 ug/1.0 mL)
1,2,4-trichlorobenzene	pentachlorophenol
acenaphthene	phenol
2,4-dinitrotoluene	2-chlorophenol
pyrene	4-chloro-3-methylphenol
N-nitroso-di-n-propylamine	4-nitrophenol
1,4-dichlorobenzene	

Prepare a spiking solution that contains each of the above in methanol. Store the spiking solutions at 4°C (+2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicate a problem.

Matrix spikes also serve as duplicates, therefore, add volume specified in Sample Extraction section to each of two 30-g portions from one sample chosen for spiking.

2.5 Low Level Sample Preparation

2.5.1 Decant and discard any water layer on a sediment sample. Mix samples thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

2.5.1.1 Transfer 50 g of soil/sediment to 100 ml beaker. Add 50 ml of water and stir for 1 hour. Determine pH of sample with glass electrode and pH meter while stirring. Report pH value on appropriate data sheets. If the pH of the soil is greater than 11 or less than 5, contact the Deputy Project Officer cited in the contract for instructions on how to handle the sample. Document the instructions in the Case Narrative. Discard this portion of sample.

2.5.2 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30 g of sample to the nearest 0.1 g into a 400-mL beaker and add 60 g of anhydrous powdered sodium sulfate. Mix well. The sample should have a sandy texture at this point. Immediately, add 100 mL of 1:1 methylene chloride - acetone to the sample, then add the surrogates according to paragraph 2.5.2.3.

2.5.2.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sediment into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\frac{\text{Percent moisture}}{\text{g of sample} - \text{g of dry sample}} \times 100 = \text{Z moisture}$$

2.5.2.2 Weigh out two 30 g (record weight to nearest 0.1 g) portions for use as matrix and matrix spike duplicates according to 2.5.2. When using GPC cleanup, add 2.0 mL of the base/neutral and acid matrix spike to each of two portions. When not using GPC cleanup, add 1.0 mL of base/neutral and acid matrix spike to each of the other two portions.

2.5.2.3 When using GPC, add 1.0 mL of base/neutral and acid surrogate standard to the sample. When not using GPC, add 0.5 mL of BNA surrogate standard to the sample.

2.5.3 Place the bottom surface of the tip of the 3/4 inch disruptor horn about 1/2 inch below the surface of the solvent but above the sediment layer.

2.5.4 Sonicate for 1 1/2 minutes with the W-385 (or 3 minutes with the W-375), using No. 208 3/4 inch standard disruptor horn with output control knob set at 10 (or No. 305 3/4 inch tapped high gain "Q" disruptor horn at 5) and mode switch on "1 sec. pulse" and Z duty cycle knob set at 50%. Do NOT use MICROTIP probe. (If using a sonicator other than Models W-375 or W-385, contact the Project Officer for appropriate output settings).

2.5.5 Decant and filter extracts through Whatman #41 filter paper using vacuum filtration or centrifuge and decant extraction solvent.

2.5.6 Repeat the extraction two more times with 2 additional 100 mL portions of 1:1 methylene chloride - acetone. Before each extraction, make certain that the sodium sulfate is free flowing and not a consolidated mass. As required, break up large lumps with a clean spatula, or very carefully with the tip of the probe. Decant off the extraction solvent after each sonication. On the final sonication, pour the entire sample into the Buchner funnel and rinse with 1:1 methylene chloride - acetone.

2.5.6.1 If the sample is to be screened from the low level method, take 5.0 mL and concentrate to 1.0 mL following paragraph 2.7.2 or 2.7.3. Note that the sample volume in this case is 5.0 mL not 10.0 mL as given in 2.7.2. Screen the extract as per Section III, paragraph 1., "Screening of Extractable Organic Extracts". Transfer the remainder of the 1 mL back to the total extract from paragraph 2.5.6 after GC/FID or GC/MS screening. (CAUTION: To minimize sample loss, autosamplers which pre-flush samples through the syringe should not be used.)

2.5.7 Transfer the extract to a Kuderna-Danish (K-D) concentrator consisting of a 10 mL concentrator tube and a 500 mL evaporative flask. Other concentration devices or techniques may be used if equivalency is demonstrated for all extractable compounds listed in Exhibit C.

2.5.8 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL methylene chloride to the top. Place the K-D apparatus on a hot water bath (80 to 90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes, and make up to 10 mL volume with methylene chloride.

2.5.9 If GPC cleanup is not used proceed to paragraph 2.7.

2.6. Extract Cleanup

2.6.1 GPC Setup and Calibration

2.6.1.1 Packing the column - Place 70 g of Bio Beads SX-3 in a 400 mL beaker. Cover the beads with methylene chloride; allow the beads to swell overnight (before packing the columns). Transfer the swelled beads to

the column and start pumping solvent through the column, from bottom to top, at 5.0 mL/min. After approximately 1 hour, adjust the pressure on the column to 7 to 10 psi and pump an additional 4 hours to remove air from the column. Adjust the column pressure periodically as required to maintain 7 to 10 psi.

2.6.1.2 Calibration of the column - Load 5 mL of the corn oil solution into sample loop No. 1 and 5 mL of the phthalatephenol solution into loop No. 2. Inject the corn oil and collect 10 mL fraction (i.e., change fraction at 2-minute intervals) for 36 minutes. Inject the phthalate-phenol solution and collect 15 mL fractions for 60 minutes. Determine the corn oil elution pattern by evaporation of each fraction to dryness followed by a gravimetric determination of the residue. Analyze the phthalate-phenol fractions by GC/FID on the DB-5 capillary column, a UV spectrophotometer, or a GC/MS system. Plot the concentration of each component in each fraction versus total eluent volume (or time) from the injection points. Choose a "dump time" which allows $\geq 85\%$ removal of the corn oil and $\geq 85\%$ recovery of the bis(2-ethylhexyl)-phthalate. Choose the "collect time" to extend at least 10 minutes after the elution of pentachlorophenol. Wash the column at least 15 minutes between samples. Typical parameters selected are: Dump time, 30 minutes (150 mL), collect time, 36 minutes (180 mL), and wash time, 15 minutes (75 mL). The column can also be calibrated by the use of a 254 nm UV detector in place of gravimetric and GC analyses of fractions. Measure the peak areas at various elution times to determine appropriate fractions.

The SX-3 Bio Beads column may be reused for several months, even if discoloration occurs. System calibration usually remains constant over this period of time if column flowrate remains constant.

2.6.2 GPC Extract Cleanup

Pre-filter or load all extracts via the filter holder to avoid particulates that might stop the flow. Load one 5.0 mL aliquot of the extract onto the GPC column. Do not apply excessive pressure when loading the GPC. Purge the sample loading tubing thoroughly with solvent between extracts. After especially dirty extracts, run a GPC blank (methylene chloride) to check for carry-over. Process the extracts using the dump, collect, and wash parameters determined from the calibration and collect the cleaned extracts in 400 mL beakers tightly covered with aluminum foil. The phthalate-phenol calibration solution shall

be taken through the cleanup cycle with each set of 23 extracts loaded into the GPC. The recovery for each compound must be $\geq 85\%$. This must be determined on a GC/FID, using a DB-5 capillary column, a UV recording spectrophotometer, or a GC/MS system. A copy of the printouts of standard and check solution are required as deliverables with each case. Show % recovery on the copy.

2.6.3 Concentrate the extract as per paragraphs 2.5.7 and 2.5.8.

2.7 Final Concentration of Extract with Optional Extract Splitting Procedure

If the extract in 2.5.8 is to be used only for semivolatile analysis, it must be concentrated to a volume of 1.0 ml, following the procedure in 2.7.2.1.

If the extract in 2.5.8 is to be used for both semivolatile and pesticide/PCB analyses, then it must be split into two portions. In that case, follow the procedure in 2.7.1 to obtain the pesticide portion, and follow that with the procedure in 2.7.2.2 to obtain the semivolatile portion.

Refer to Exhibit D PEST for specific instructions regarding the treatment of extracts for pesticide analysis.

2.7.1 If the same extract is used for both semivolatile and pesticide/PCB analyses, to split out the pesticide extract, transfer 0.5 mL of the 10 mL methylene chloride extract from 2.5.8 to a separate concentrator tube. Add 5 mL of hexane and a silicon carbide boiling chip and mix using vortex mixer. Attach a two-ball micro-Snyder column. Pre-wet the Snyder column by adding 0.5 mL of hexane to the top of the column. Place the K-D apparatus on a hot water bath (80°-90°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. Concentrate the extract to an apparent volume of less than 1 mL. Use Nitrogen blowdown (see 2.7.3) to reduce the volume to 0.5 mL. Add 0.5 mL of acetone. The pesticide extract must now be passed through an alumina column to remove the BNA surrogates and polar interferences. Proceed to paragraph 2.8 of the pesticide/PCB method (Exhibit D PEST).

2.7.2 Concentration of the semivolatile extract.

2.7.2.1 If the extract in 2.5.8 was not split to obtain a portion for pesticide analysis, reattach the micro-Snyder column to the concentrator tube used in 2.5.8 which contains the 10 mL extract and add a fresh silicon carbide boiling chip to the concentrator tube. Pre-wet the Snyder column with 0.5 mL of

methylene chloride. Place the K-D apparatus on the hot water bath (80°-90°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. When the apparent volume of the liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column and rinse the lower joint into the concentrator tube with 0.2 mL of methylene chloride. Adjust the final volume to 1.0 mL with methylene chloride. If GPC cleanup was used, this 1.0 mL represents a twofold dilution to account for only half of the extract going through the GPC.

- 2.7.2.2 If the extract in 2.5.8 was split in 2.7.1 to obtain a portion for pesticide analysis, reattach the micro-Snyder column to the concentrator tube used in 2.5.8 which contains the 9.5 mL extract and add a fresh silicon carbide boiling chip to the concentrator tube. Pre-wet the Snyder column with 0.5 mL of methylene chloride. Place the K-D apparatus on the hot water bath (80°-90°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. When the apparent volume of the liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column and rinse the lower joint into the concentrator tube with 0.2 mL of methylene chloride. Adjust the final volume to 0.95 mL with methylene chloride. If GPC cleanup was used, this 0.95 mL represents a twofold dilution to account for only half of the extract going through the GPC, and therefore, the sample detection limit for the sample would be 2x CRQL (see Exhibit B).

- 2.7.3 Nitrogen blowdown technique (taken from ASTM Method D 3086). The following method may be used for final concentration of the BNA extract instead of the procedures in paragraph 2.7.2. Place the concentrator tube in a warm water bath (35°C) and evaporate the solvent volume to below 1 mL using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). Caution: New plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.

The internal wall of the tube must be rinsed down several times with methylene chloride during the operation. During evaporation, the tube solvent level must be kept below the water level of the bath. The extract must never be allowed to become dry.

If the extract in 2.5.8 was not split for both semivolatile and pesticide analyses, bring the final volume of the extract to 1.0 mL with methylene chloride. This represents a ten-fold concentration. If the extract in 2.5.8 was split in 2.7.1, then bring the final volume of the semivolatile portion to 0.95 mL with methylene chloride. This represents a similar ten-fold concentration. In either case, if GPC cleanup techniques were employed, the final volume (1.0 or 0.95 mL) represents a two-fold dilution to account for the fact that only half the extract went through the GPC.

- 2.7.4 Store all extracts at 4°C (+2°C) in the dark in Teflon-sealed containers.

SECTION III

SCREENING OF SEMIVOLATILE.

ORGANIC EXTRACTS

1. Summary of Method

- 1.1 The solvent extracts of water and sediment/soil are screened on a gas chromatograph/flame ionization detector (GC/FID) using a fused silica capillary column (FSCC). The results of the screen will determine the concentration of extract taken for GC/MS analysis.

2. Apparatus and Materials

- 2.1 Gas chromatograph - An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port must be designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

2.1.1 Above GC equipped with flame ionization detector.

2.1.2 GC column - 30 m x 0.32 mm, 1 micron film thickness, silicone coated, fused silica capillary column (J & W Scientific DB-5 or equivalent).

3. Reagents

3.1 Methylene chloride - pesticide residue analysis grade or equivalent.

3.2 GC calibration standard. Prepare a standard solution containing phenol, phenanthrene, and di-n-octylphthalate.

3.2.1 Stock standard solutions (1.00 ug/uL)-Stock standard solutions can be prepared from pure standard materials or purchased solutions.

3.2.1.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality methylene chloride and dilute to volume in a 10 mL volumetric flask. Larger volumes may be used at the convenience of the analyst. If compound purity is assayed at 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source and are traceable to EMSL/LV-supplied standards.

3.2.1.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at -10°C to -20°C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Stock standard

solutions must be replaced after six months or sooner if comparison with quality control check samples indicates a problem. Standards prepared from gases - or reactive compounds such as styrene must be replaced after two months, or sooner if comparison with quality control check samples indicates a problem.

- 3.2.2 Prepare a working standard mixture of the three compounds in methylene chloride. The concentration must be such that the volume injected equals 50 ng of each compound. The storage and stability requirements are the same as specified in 3.2.1.2.

4. GC Calibration

- 4.1 At the beginning of each 12 hour shift, inject the GC calibration standard. The following criteria must be:

- 4.1.1 Standardized for half scale response from 50 ng of phenanthrene.
- 4.1.2 Adequately separates phenol from the solvent front.
- 4.1.3 Minimum of quarter scale response for 50 ng of di-n-octylphthalate.

5. GC/FID Screening

- 5.1 Suggested GC operating conditions:

Initial Column Temperature Hold - 50°C for 4 minutes
 Column Temperature Program - 50 - 280°C at 8 degrees/min.
 Final Column Temperature Hold - 280°C for 8 minutes
 Injector - Grob-type, splitless
 Sample Volume - 1 μ L - 2 μ L
 Carrier Gas - Helium at 30 cm^3/sec

- 5.2 Inject the GC calibration standard and ensure the criteria specified in 4. are met before injecting samples. Estimate the response for 10 ng of phenanthrene.

- 5.3 Inject the appropriate extracts from Section II, including blanks.

6. Interpretation of Chromatograms

- 6.1 Water

- 6.1.1 If no sample peaks are detected, or all are less than full scale deflection, the undiluted extract is analyzed on GC/MS.

- 6.1.2 If any sample peaks are greater than full scale deflection, calculate the dilution necessary to reduce the major peaks to between half and full scale deflection. Use this dilution factor to dilute the extract for GC/MS analysis.

6.2 Soil/Sediment

- 6.2.1 If no sample peaks from the extract (from low or medium level preparation) are detected, or all are less than 10% full scale deflection, the sample must be prepared by the low level protocol, Section II, Part C, paragraph 2.
- 6.2.2 Peaks are detected at greater than 10% full scale deflection and less than or equal to full scale deflection.
- 6.2.2.1 If the screen is from the medium level extract, proceed with GC/MS analysis of this extract with appropriate dilution if necessary.
- 6.2.2.2 If screen is from the low level extract, discard extract and prepare sample by medium level method for GC/MS analysis.
- 6.2.3 Peaks are detected at greater than full scale deflection:
- 6.2.3.1 If the screen is from the medium level preparation, calculate the dilution necessary to reduce the major peaks to between half and full scale deflection. Use this dilution factor to dilute the extract. This dilution is analyzed by GC/MS for extractable organics.
- 6.2.3.2 If the screen is from the low level preparation, discard the extract and prepare a sample by the medium level method for GC/MS analysis.

7. GC/MS Analysis

- 7.1 Use the information from 6. to perform the GC/MS analysis of extractables in Section IV, GC/MS Analysis of Semivolatiles, paragraph 1.

SECTION IV

GC/MS ANALYSIS OF SEMIVOLATILES

1. Summary of Method

This method is to be used for the GC/MS analysis of semivolatiles screened by Section III protocols and for confirmation of pesticides/PCBs identified by GC/EC, if concentrations permit.

2. Apparatus and Materials

2.1 Gas chromatograph/mass spectrometer system.

- 2.1.1 Gas chromatograph - An analytical system complete with a temperature programmable gas chromatograph suitable for splitless injection and all required accessories including syringes, analytical columns, and gases.!
- 2.1.2 Column - 30 m x 0.25 mm ID (or 0.32 mm) bonded-phase silicone coated fused silica capillary column (J&W Scientific DB-5 or equivalent). A film thickness of 1.0 micron is recommended because of its larger capacity. A film thickness of 0.25 micron may be used.
- 2.1.3 Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all required criteria when 50 ng of decafluorotriphenylphosphine (DFTPP) is injected through the GC inlet.

NOTE: DFTPP criteria must be met before any sample extracts are analyzed. Any samples analyzed when DFTPP criteria have not been met will require reanalysis at no cost to the Government.
- 2.1.4 Data system - A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

3. Reagents

- 3.1 Internal standards - 1,4 dichlorobenzene-d₄, naphthalene-d₈, acenaphthene-d₁₀, phenanthrene-d₁₀, chrysene-d₁₂, perylene-d₁₂. An internal standard solution can be prepared by dissolving

200 mg of each compound in 50 mL of methylene chloride. It may be necessary to use 5 to 10 percent benzene or toluene in this solution and a few minutes of ultrasonic mixing in order to dissolve all the constituents. The resulting solution will contain each standard at a concentration of 4000 ng/uL. A 10 uL portion of this solution should be added to each 1 mL of sample extract. This will give a concentration of 40 ng/uL of each constituent.

- 3.2 Prepare calibration standards at a minimum of five concentration levels. Each calibration standard should contain each compound of interest and each surrogate standard. See GC/MS calibration in Exhibit E for calibration standard concentration.

Great care must be taken to maintain the integrity of all standard solutions. Store all standard solutions at -10°C to -20°C in screw-cap amber bottles with teflon liners. Fresh standards should be prepared every twelve months at a minimum. The continuing calibration standard should be prepared weekly and stored at 4°C (+2°C).

4. Calibration

- 4.1 Each GC/MS system must have the hardware tuned to meet the criteria listed in Exhibit E for a 50 ng injection of decafluorotriphenyl phosphine (DFTPP). No sample analyses can begin until all these criteria are met. This criteria must be demonstrated each 12 hour shift. DFTPP has to be injected to meet this criterion. Post-acquisition manipulation of abundances is not acceptable.
- 4.2 The internal standards selected in paragraph 2.3.1 should permit most components of interest in a chromatogram to have retention times of 0.80 to 1.20 relative to the internal standards (see instructions for Form VI, Initial Calibration Data). Use the base peak ion from the specific internal standard as the primary ion for quantification, found in Exhibit E, Table 2.2. If interferences are noted, use the next most intense ion as the secondary ion, i.e. For 1,4-dichlorobenzene-d₄ use m/z 152 for quantification.
- 4.2.1 The internal standards are added to all calibration standards and all sample extracts just prior to analysis by GC/MS. A 10 uL aliquot of the internal standard solution should be added to a 1 mL aliquot of calibration standards.
- 4.3 Analyze 1 uL of each calibration standard and tabulate the area of the primary characteristic ion against concentration for each compound including the surrogate compounds. Calculate relative response factors (RRF) for each compound using Equation 1.

$$RRF = \frac{A_x}{A_{IS}} \times \frac{C_{IS}}{C_x}$$

Equation 1.

Where:

- A_x = Area of the characteristic ion for the compound to be measured.
- A_{is} = Area of the characteristic ion for the specific internal standard from Exhibit E.
- C_{is} = Concentration of the internal standard (ng/uL).
- C_x = Concentration of the compound to be measured (ng/uL).

- 4.3.1 The average relative response factor (RRF) should be calculated for all compounds. A system performance check must be made before this calibration curve is used. Four compounds (the system performance check compounds) are checked for a minimum average relative response factor. These compounds (the SPCC) are N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, 4-nitrophenol. See instructions in Exhibit E for Form VI, Initial Calibration Data for more details.
- 4.3.2 A % Relative Standard Deviation (XRSD) is calculated for thirteen compounds labeled the Calibration Check Compounds (CCC) on Form VI SV and in Table 2.3, Exhibit E, III SV. A maximum % RSD is also specified for these compounds. These criteria must be met for the calibration curve to be valid.
- 4.4 A check of the calibration curve must be performed once every 12 hours during analysis. These criteria are described in detail in the instructions for Form VII, Calibration Check. The minimum relative response factor for the system performance check compounds must be checked. If this criteria is met, the relative response factors of all compounds are calculated. A percent difference of the daily (12 hour) relative response factor compared to the average relative response factor from the initial curve is calculated. A maximum percent difference is allowed for each compound flagged as 'CCC' on Form VII. Only after both these criteria are met can sample analysis begin.
- 4.5 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each standard. If EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

5. GC/MS Analysis

- 5.1 The following instrumental parameters are required for all performance tests and for all sample analyses:

Electron Energy - 70 volts (nominal)
Mass Range - 35 to 500 amu
Scan Time - not to exceed 1 second per scan

- 5.2 Combine 0.5 mL of the base/neutral extract and 0.5 mL of acid from the water extract prior to analysis.
- 5.3 Internal standard solution is added to each sample extract. For water and/or medium soil extracts, add 10 μ L of internal standard solution to each accurately measured 1.0 mL of sample extract. If the low soil extracts required a pesticide split (see Section II, Part C, paragraph 2.7), add 9.5 μ L of internal standard solution to each accurately measured 0.95 mL of sample extract.

Analyze the 1.0 mL extract by GC/MS using a bonded-phase silicone-coated fused silica capillary column. The recommended GC operating conditions to be used are as follows:

Initial Column Temperature Hold - 40°C for 4 minutes
Column Temperature Program - 40-270°C at 10 degrees/min.
Final Column Temperature Hold - 270°C for 10 minutes
Injector Temperature - 250-300°C
Transfer Line Temperature - 250-300°C
Source Temperature - according to manufacturer's specifications

Injector-Grob-type, splitless
Sample Volume - 1 - 2 μ L
Carrier Gas - Helium at 30 cm^3/sec

NOTE: Make any extract dilution indicated by characterization prior to the addition of internal standards. If any further dilutions of water or soil/sediment extracts are made, additional internal standards must be added to maintain the required 40 ng/ μ L of each constituent in the extract volume. If the concentration of any compound exceeds the initial calibration range, the extract must be diluted and reanalyzed. See Exhibit E, Section III, SV, Part 6. Secondary ion quantitation is only allowed when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the Case Narrative.

6. Qualitative Analysis

- 6.1 The compounds listed in the Target Compound List (TCL), Exhibit C, shall be identified by an analyst competent in the interpretation

of mass spectra (see PreAward Bid Confirmation description) by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications: (1) elution of the sample component at the GC relative retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

- 6.1.1 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.
- 6.1.2 For comparison of standard and sample component mass spectra, mass spectra obtained on the contractor's GC/MS are required. Once obtained, these standard spectra may be used for identification purposes, only if the contractor's GC/MS meets the DFTPP daily tuning requirements. These standard spectra may be obtained from the run used to obtain reference RRTs.
- 6.1.3 The requirements for qualitative verification by comparison of mass spectra are as follows:
 - 6.1.3.1 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.
 - 6.1.3.2 The relative intensities of ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)
 - 6.1.3.3 Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. In Task III, the verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the CRQL report the actual value followed by "J", e.g. "3J."

- 6.1.4 If a compound cannot be verified by all of the criteria in 6.1.3, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the Contractor shall report that identification and proceed with quantification in 7.
- 6.2 A library search shall be executed for non-TCL sample components for the purpose of tentative identification. For this purpose, the 1985 release of the National Bureau of Standards Mass Spectral Library (or more recent release), containing 42,261 spectra, shall be used.
- 6.2.1 Up to 20 nonsurrogate organic compounds of greatest apparent concentration not listed in Exhibit C for the combined base/neutral/acid fraction shall be tentatively identified via a forward search of the NBS mass spectral library. (Substances with responses less than 10% of the nearest internal standard are not required to be searched in this fashion). Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. NOTE: Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.
- 6.2.2 Guidelines for making tentative identification:
- 6.2.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- 6.2.2.2 The relative intensities of the major ions should agree within + 20%. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.
- 6.2.2.3 Molecular ions present in reference spectrum should be present in sample spectrum.
- 6.2.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- 6.2.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. NOTE: Data system library reduction programs can sometimes create these discrepancies.

- 6.2.3 If in the technical judgement of the mass interpretation spectral specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

7. Quantitation

- 7.1 TCL components identified shall be quantified by the internal standard method. The internal standard used shall be the one nearest the retention time to that of a given analyte (see Exhibit E, Tables 2.1 and 2.2). The EICP area of characteristic ions of analytes listed in Tables 4, 5 and 6 are used.

Internal standard responses and retention times in all samples must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate. The criteria are described in detail in the instructions for Form VIII, Internal Standard Area Summary. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. If the analysis of a subsequent sample or standard indicates that the system is functioning properly, then corrections may not be required. The samples or standards with EICP areas outside the limits must be re-analyzed, and treated according to 7.1.1 and 7.1.2 below. If corrections are made, then the laboratory must demonstrate that the mass spectrometric system is functioning properly. This must be accomplished by the analysis of a standard or sample that does meet the EICP criteria. After corrections are made, the re-analysis of samples analyzed while the system was malfunctioning is required.

- 7.1.1 If after re-analysis, the EICP areas for all internal standards are inside the contract limits (-50% to +100%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, only submit data from the analysis with EICP's within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.
- 7.1.2 If the re-analysis of the sample does not solve the problem, i.e., the EICP areas are outside the contract limits for both analyses, then submit the EICP data and sample data from both analyses. Distinguish between the initial analysis and the re-analysis on all data deliverables, using the sample suffixes specified in Exhibit B. Document in the Case Narrative all inspection and corrective actions taken.

- 7.2 The relative response factor (RRF) from the daily standard analysis is used to calculate the concentration in the sample. Secondary ions may be used if interferences are present. The area of a secondary ion cannot be substituted for the area of a primary ion unless a relative response factor is calculated using the secondary ion. When TCL Compounds are below contract required quantitation limits (CRQL) but the spectra meets the identification criteria, report the concentration with a "J." For example, if CRQL is 10 ug/L and concentration of 3 ug/L is calculated, report as "3J."

- 7.2.1 Calculate the concentration in the sample using the relative response factor (RRF) as determined in paragraph 4.3 and the following equation:

Water

$$\text{Concentration ug/L} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(V_o)(V_i)}$$

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard injected in nanograms (ng)

V_o = Volume of water extracted in milliliters (mL)

V_i = Volume of extract injected (uL)

V_t = Volume of total extract
(Use 2000 uL or a factor of this when dilutions are made. The 2,000 uL is derived from combining half of the 1 mL BN extract and half of the 1 mL A extract.)

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Soil/Sediment

$$\text{Concentration ug/kg} = \frac{(A_x)(I_s)(V_r)}{(A_{1s})(RRF)(V_1)(W_s)(D)}$$

(Dry weight basis)

Where:

A_x, I_s, A_{1s} = Same as given for water, above

V_t = Volume of low level total extract
(Use 1000 uL or a factor of this when dilutions are made. If GPC cleanup is used, the volume is 2,000 uL. The 1000 uL is derived from concentrating the 9.5 mL extract to 0.95 mL.)

- or - V_t = Volume of medium level extract
(Use 2,000 uL or a factor of this when dilutions are made. The 2,000 uL is derived from concentrating 5 mL of the 10 mL extract to 1 mL.)

V_1 = Volume of extract injected (uL)

D = $\frac{100 - \% \text{ moisture}}{100}$

W_s = Weight of sample extracted (grams)

7.3 An estimated concentration for non-TCL components tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard free of interferences shall be used.

7.3.1 The formula for calculating concentrations is the same as in paragraph 7.2.1. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A relative response factor (RRF) of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

7.4 Calculate surrogate standard recovery on all samples, blanks and spikes. Determine if recovery is within limits and report on appropriate form.

7.4.1 If recovery is not within limits (i.e., if two surrogates from either base/neutral or acid fractions are out of limits or if recovery of any one surrogate in either fraction is below 10%), the following is required.

o Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

o Reanalyze the sample if none of the above reveal a problem.

- 7.4.2 If the reanalysis of the sample solves the problem, then the problem was within the laboratory's control. Therefore, only submit data from the analysis with surrogate spike recoveries within the contract windows. This shall be considered the initial analysis and shall be reported as such on all data deliverables.
- 7.4.3 If none of the steps in 7.4.1 or 7.4.2 solve the problem, then reextract and reanalyze the sample. If the reextraction and reanalysis of the sample solves the problem, then the problem was within the laboratory's control. Therefore, only submit data from the analysis with surrogate spike recoveries within the contract windows. This shall be considered the initial analysis and shall be reported as such on all data deliverables.
- 7.4.4 If the reextraction and reanalysis of the sample does not solve the problem, i.e., the surrogate recoveries are outside the contract limits for both analyses, then submit the surrogate spike recovery data and the sample analysis data from analysis of both sample extracts. Distinguish between the initial analysis and the reanalysis on all data deliverables, using the sample suffixes specified in Exhibit B.
- 7.4.5 If the sample with surrogate recoveries outside the limits is the sample used for the matrix spike and matrix spike duplicate and the surrogate recoveries of the matrix spike and matrix spike duplicate show the same pattern (i.e., outside the limits), then the sample, matrix spike, and matrix spike duplicate do not require re-analysis. Document in the narrative the similarity in surrogate recoveries.

Table 4.
Characteristic Ions for Semivolatile TCL Compounds

Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(-2-Chloroethyl)Ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
Benzyl Alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
bis(2-chloroisopropyl)Ether	45	77, 79
4-Methylphenol	108	107
N-Nitroso-Di-Propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
Benzoic Acid	122	105, 77
bis(-2-Chloroethoxy)Methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-Methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethyl Phthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141

(continued)

Table 4. (continued)
Characteristic Ions for Semivolatile TCL Compounds

Parameter	Primary Ion	Secondary Ion(s)
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-Methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Di-N-Butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)Anthracene	228	229, 226
bis(2-Ethylhexyl)Phthalate	149	167, 279
Chrysene	228	226, 229
Di-N-Octyl Phthalate	149	-
Benzo(b)Fluoranthene	252	253, 125
Benzo(k)Fluoranthene	252	253, 125
Benzo(a)Pyrene	252	253, 125
Indeno(1,2,3-cd)Pyrene	276	138, 227
Dibenz(a, h)Anthracene	278	139, 279
Benzo(g, h, i)Perylene	276	138, 277

Table 5.
Characteristic Ions for Pesticides/PCBs

Parameter	Primary Ion	Secondary Ion(s)
Alpha-BHC	183	181, 109
Beta-BHC	181	183, 109
Delta-BHC	183	181, 109
Gamma-BHC (Lindane)	183	181, 109
Heptachlor	100	272, 274
Aldrin	66	263, 220
Heptachlor Epoxide	353	355, 351
Endosulfan I	195	339, 341
Dieldrin	79	263, 279
4,4'-DDE	246	248, 176
Endrin	263	82, 81
Endosulfan II	337	339, 341
4,4'-DDD	235	237, 165
Endosulfan Sulfate	272	387, 422
4,4'-DDT	235	237, 165
Methoxychlor	227	228
Chlordane (alpha and/or gamma)	373	375, 377
Toxaphene	159	231, 233
Arochlor-1016	222	260, 292
Arochlor-1221	190	222, 260
Arochlor-1232	190	222, 260
Arochlor-1242	222	256, 292
Arochlor-1248	292	362, 326
Arochlor-1254	292	362, 326
Arochlor-1260	360	362, 394
Endrin Ketone	317	67, 319

Table 6.
Characteristic Ions for Surrogates and
Internal Standards for Semivolatile Compounds

SURROGATES	Primary Ion	Secondary Ion(s)
Phenol-d ₅	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
d-5 Nitrobenzene	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl	244	122, 212
<u>INTERNAL STANDARDS</u>		
1,4-Dichlorobenzene-d ₄	152	115
Naphthalene-d ₈	136	68
Acenaphthene-d ₁₀	164	162, 160
Phenanthrene-d ₁₀	188	94, 80
Chrysene-d ₁₂	240	120, 236
Perylene-d ₁₂	264	260, 265

SECTION III SV

SEMIVOLATILES QA/QC

REQUIREMENTS

This Section outlines the minimum quality control (QC) operations necessary to satisfy the analytical requirements associated with the determination of semi-volatile organic TCL compounds in water and soil/sediment samples. These QC operations are as follows:

- o Documentation of GC/MS Mass Calibration and Abundance Pattern
- o Documentation of GC/MS Response Factor Stability
- o Internal Standard Response and Retention Time Monitoring
- o Method Blank Analysis
- o Surrogate Spike Response Monitoring
- o Matrix Spike and Matrix Spike Duplicate Analysis

PART 1 - TUNING AND GC/MS MASS CALIBRATION

1. Summary

It is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria prior to initiating any on-going data collection. This is accomplished through the analysis of Decafluorotriphenylphosphine (DFTPP).

Definition: The twelve (12) hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that the laboratory submits as documentation of a compliant tune. The time period ends after twelve (12) hours has elapsed according to the system clock.

1.1 Decafluorotriphenylphosphine (DFTPP)

- 1.1.1 Each GC/MS system used for the analysis of semivolatile or pesticide TCL compounds must be hardware tuned to meet the abundance criteria listed in Table 1.2 for a 50 ng injection of decafluorotriphenylphosphine (DFTPP). DFTPP may be analyzed separately or as part of the calibration standard. The criteria must be demonstrated daily or for each twelve (12) hour period, whichever is more frequent, before samples can be analyzed. DFTPP must be injected to meet this criterion. If required, background subtraction must be straightforward and designed only to eliminate column bleed or instrument background ions. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract specifications are unacceptable. NOTE: All instrument conditions must be identical to those used in sample analysis, except that a different temperature program may be used.

- 1.1.2 Whenever the contractor takes corrective action which may change or affect the tuning criteria for DFTPP (e.g., ion source cleaning or repair, etc.), the tune must be verified irrespective of the 12-hour tuning requirements.

TABLE 1.2. DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
70	less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

1.2 Documentation

The contractor shall provide documentation of the calibration in the form of a bar graph spectrum and as a mass listing.

- 1.2.1 The contractor shall complete a Form V (GC/MS Tuning and Mass Calibration) each time an analytical system is tuned. In addition, all samples, standards, blanks, matrix spikes, and matrix spike duplicates analyzed during a particular tune must be summarized in chronological order on the bottom of the appropriate Form V. Detailed instructions for the completion of Form V are found in Exhibit B, Section III.

PART 2 - CALIBRATION OF THE GC/MS SYSTEM

2. Summary

Prior to the analysis of samples and required blanks and after tuning criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing TCL compound standards. Once the system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

- 2.1 Prepare calibration standards as described in Exhibit D SV, Section IV, to yield the following specific concentrations:

2.1.1 Semivolatile TCL Compounds

Initial calibration of semivolatile TCL compounds is required at 20, 50, 80, 120, and 160 total nanograms. If an analyte saturates at the 160 total nanogram concentration level, and the GC/MS system is calibrated to achieve a detection sensitivity of no less than the CRQL, the laboratory must document it on Form VI and in the Case narrative, and attach a quantitation report and RIC. In this instance, the laboratory should calculate the results based on a four-point initial calibration for the specific analyte. The use of a secondary ion for quantitation is only allowed when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the Case Narrative. Nine compounds: Benzoic Acid, 2,4-Dinitrophenol, 2,4,5-Trichlorophenol, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, 4-Nitrophenol, 4,6-Dinitro-2-Methylphenol, and Pentachlorophenol will only require a four-point initial calibration at 50, 80, 120, and 160 total nanograms since detection at less than 50 nanograms per injection is difficult.

- 2.2 The USEPA plans to develop performance based criteria for response factor data acquired during this program. To accomplish this goal, the Agency has specified both the concentration levels for initial calibration and has also specified the specific internal standard to be used on a compound-by-compound basis for quantitation (Table 2.2). Establishment of standard calibration procedures is necessary and deviations by the contractor will not be allowed.
- 2.3 Analyze each calibration standard and tabulate the area of the primary characteristic ion (Exhibit D SV, Table 4) against concentration for each compound including all contract required surrogate compounds. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units. Late eluting compounds usually will have much better agreement.

Using Table 2.2, calculate the relative response factors (RRF) for each compound at each concentration level using Equation 2.1.

$$RRF = \frac{A_x}{A_{IS}} \times \frac{C_{IS}}{C_x} \quad \text{Eq. 2.1}$$

where,

A_x = Area of the characteristic ion for the compound to be measured.

A_{IS} = Area of the characteristic ion for the specific internal standards from Table 2.1 or 2.2.

C_{IS} = Concentration of the internal standard (ng/uL).

C_x = Concentration of the compound to be measured (ng/uL).

- 2.3.1 Using the relative response factors (RRF) from the initial calibration, calculate the percent relative standard deviations (ZRSD) for compounds labeled on Form VI as Calibration Check Compounds and shown in Table 2.3 (see 2.6.2) using Equation 2.2.

$$\text{ZRSD} = \frac{\text{SD}}{\bar{x}} \times 100 \quad \text{Eq. 2.2}$$

where,

RSD = Relative Standard Deviation

SD = Standard Deviation of initial response factors (per compound)

$$\text{where: SD} = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N-1}}$$

\bar{x} = mean of initial relative response factors (per compound)

The ZRSD for each individual Calibration Check Compound must be less than or equal to 30.0 percent. This criteria must be met for the initial calibration to be valid.

- 2.4 A system performance check must be performed to ensure that minimum average relative response factors are met before the calibration curve is used.

2.4.1 For semivolatiles, the System Performance Check Compounds (SPCC's) are: N-Nitroso-Di-n-Propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol and 4-Nitrophenol. The minimum acceptable average relative response factor (RRF) for these compounds is 0.050. SPCC's typically have very low RRFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. These compounds are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

2.4.2 The initial calibration is valid only after both the ZRSD for CCC compounds and the minimum RRF for SPCC have been met. Only after both these criteria are met can sample analysis begin.

2.5 Documentation

Once the initial calibration is validated, calculate and report the average relative response factor (RRF) and percent relative standard deviation (ZRSD) for all TCL compounds. The contractor shall complete and submit Form V (the GC/MS tune for the initial calibration) and Form VI (Initial Calibration Data) for each instrument used to analyze samples under this protocol. Detailed instructions for completion of Form VI are in Exhibit B, Section III.

TABLE 2.2. SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING TCL ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d ₄	Naphthalene-d ₈	Acenaphthene-d ₁₀	Phenanthrene-d ₁₀	Chrysene-d ₁₂	Perylene-d ₁₂
Phenol	Nitrobenzene	Hexachlorocyclo-	4,6-Dinitro-2-	Pyrene	Di-n-octyl
bis(2-Chloroethyl)	Isophorone	pentadiene	methylphenol	Butylbenzyl	Phthalate
ether	2-Nitrophenol	2,4,6-Trichloro-	N-nitrosodi-	Phthalate	Benzo(b)fluor-
2-Chlorophenol	2,4-Dimethyl-	phenol	phenylamine	3,3'-Dichloro-	anthene
1,3-Dichlorobenzene	phenol	2,4,5-Trichloro-	1,2-Diphenylhy-	benzidine	Benzo(k)fluor-
1,4-Dichlorobenzene	Benzoic acid	phenol	drazine	Benzo(a)-	anthene
Benzyl Alcohol	bis(2-Chloro-	2-Chloronaphthalene	4-Bromophenyl	anthracene	Benzo(a)pyrene
1,2-Dichlorobenzene	ethoxy)methane	2-Nitroaniline	Phenyl Ether	bis(2-ethylhexyl)	Indeno(1,2,3-cd)
2-Methylphenol	2,4-Dichloro-	Dimethyl Phthalate	Hexachloro-	Phthalate	pyrene
bis(2-Chloroiso-	phenol	Acenaphthylene	benzene	Chrysene	Dibenz(a,h)
propyl)ether	1,2,4-Trichloro-	3-Nitroaniline	Pentachloro-	Terphenyl-d ₁₄	anthracene
4-Methylphenol	benzene	Acenaphthene	phenol	(surr)	Benzo(g,h,i)
N-nitroso-Di-n-	Naphthalene	2,4-Dinitrophenol	Phenanthrene		perylene
propylamine	4-Chloroaniline	4-Nitrophenol	Anthracene		
Hexachloroethane	Hexachloro-	Dibenzofuran	Di-n-butyl		
2-Fluorophenol	butadiene	2,4-Dinitrotoluene	Phthalate		
(surr)	4-Chloro-3-	2,6-Dinitrotoluene	Fluoranthene		
Phenol-d ₆ (surr)	methylphenol	Diethyl Phthalate			
	2-Methylnaphth-	4-Chlorophenyl-			
	alene	phenyl ether			
	Nitrobenzene-d ₅	Fluorene			
	(surr)	4-Nitroaniline			
		2-Fluorobiphenyl			
		(surr)			
		2,4,6-Tribromo			
		Phenol (surr)			

Surr = surrogate compound

2.6 Continuing Calibration

A calibration standard(s) containing all semivolatile TCL compounds, including all required surrogates, must be analyzed each twelve hours during analysis (see definition of twelve hour time period, paragraph 1. of this Section). Compare the relative response factor data from the standards each twelve hours with the average relative response factor from the initial calibration for a specific instrument. A system performance check must be made each twelve hours. If the SPCC criteria are met, a comparison of relative response factors is made for all compounds. This is the same check that is applied during the initial calibration (Form VI). If the minimum relative response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins.

2.6.1 Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatography system. This check must be met before analysis begins. The minimum relative response factor (RRF) for semivolatile System Performance Check Compounds (SPCC) is 0.050.

2.6.2 Calibration Check Compounds (CCC)

After the system performance check is met, Calibration Check Compounds listed in Table 2.3 are used to check the validity of the initial calibration. Calculate the percent difference using Equation 2.3.

$$\% \text{ Difference} = \frac{\overline{\text{RRF}}_I - \text{RRF}_C}{\overline{\text{RRF}}_I} \times 100 \quad \text{Eq. 2.3}$$

where,

$\overline{\text{RRF}}_I$ = average response factor from initial calibration.

RRF_C = response factor from current verification check standard.

2.6.2.1 If the percent difference for any compound is greater than 20%, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than or equal to 25.0%, the initial calibration is assumed to be valid. If the criteria are not met (>25.0% difference), for any one calibration check compound, corrective action MUST be taken. Problems similar to those listed under SPCC could affect this criteria. If no source of the problem can be determined after corrective action has been taken, a new initial five point calibration MUST be generated. These criteria MUST be met before sample analysis begins.

TABLE 2.3. CALIBRATION CHECK COMPOUNDS

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-Methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-n-phenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

2.6.3 Concentration Levels for Continuing Calibration Check

The USEPA plans to evaluate the long term stability of response factors during this program. Standardization among contract laboratories is necessary to reach these long term goals. Along with contract specified concentrations for initial calibration, the USEPA is requiring specific concentrations for each continuing calibration standard(s).

2.6.3.1 The concentration for each semivolatile TCL compound in the continuing calibration standard(s) is 50 total nanograms for all compounds.

2.7 Documentation

The contractor shall complete and submit a Form VII for each GC/MS system utilized for each twelve hour time period. Calculate and report the relative response factor and percent difference (ZD) for all compounds. Ensure that the minimum RRF for semivolatile SPCC's is 0.050. The percent difference (ZD) for each CCC compound must be less than or equal to 25.0 percent. Additional instructions for completing Form VII are found in Exhibit B, Section III.

PART 3 - METHOD BLANK ANALYSIS

3. Summary

A method blank is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples, carried through the entire analytical scheme (extraction, concentration, and analysis). For soil/sediment samples, a solid matrix suitable for semivolatile analyses is available from EMSL/LV. The method blank volume or weight must be approximately equal to the sample volumes or sample weights being processed.

3.1 Method blank analysis must be performed at the following frequency.

3.1.1 For the analysis of semivolatile TCL compounds, a method blank analysis must be performed once:

- o each Case, OR
- o each 14 calendar day period during which samples in a Case are received (said period beginning with the receipt of the first sample in that Sample Delivery Group), OR
- o each 20 samples in a Case that are of similar matrix (water or soil) or similar concentration (soil only), OR
- o whenever samples are extracted by the same procedure (separatory funnel, continuous liquid-liquid extraction, or sonication),

whichever is most frequent, on each GC/MS or GC system used to analyze samples.

3.2 It is the contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be minimized.

3.2.1 For the purposes of this protocol, an acceptable laboratory method blank should meet the criteria of paragraphs 3.2.1.1 and 3.2.1.2.

3.2.1.1 A method blank for semivolatile analysis must contain less than or equal to five times (5X) the Contract Required Quantitation Limit (CRQL from Exhibit C) of the phthalate esters in the TCL.

3.2.1.2 For all other TCL compounds not listed above, the method blank must contain less than or equal to the Contract Required Quantitation Limit of any single TCL analyte.

3.2.2 If a laboratory method blank exceeds these criteria, the contractor must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) MUST be reextracted and reanalyzed at no additional cost to the Agency. The Laboratory Manager, or his designee, must address problems and solutions in the Case Narrative (Exhibit B).

3.3 Documentation

The contractor shall report results of method blank analysis using the Organic Analysis Data Sheet (Form I) and the form for tentatively identified compounds (Form I, TIC). In addition, the samples associated with each method blank must be summarized on Form IV (Method Blank Summary). Detailed instructions for the completion of these forms are in Exhibit B, Section III.

3.3.1 The Contractor shall report ALL sample concentration data as UNCORRECTED for blanks.

PART 4 - SURROGATE SPIKE (SS) ANALYSIS**4. Summary**

Surrogate standard determinations are performed on all samples and blanks. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction in order to monitor preparation and analysis of samples.

- 4.1 Each sample, matrix spike, matrix spike duplicate, and blank are spiked with surrogate compounds prior to extraction. The surrogate spiking compounds shown in Table 4.1 are used to fortify each sample, matrix spike, matrix spike duplicate, and blank with the proper concentrations. Performance based criteria are generated from laboratory results. Therefore, deviations from the spiking protocol will not be permitted.

TABLE 4.1. SURROGATE SPIKING COMPOUNDS

Compounds	Fraction	Amount in Sample Extract* (before any optional dilutions)	
		Water	Low/Medium Soil
Nitrobenzene-d ₅	BNA	50 ug	50 ug
2-Fluorobiphenyl	BNA	50 ug	50 ug
p-Terphenyl-d ₁₄	BNA	50 ug	50 ug
Phenol-d ₅	BNA	100 ug	100 ug
2-Fluorophenol	BNA	100 ug	100 ug
2,4,6-Tribromophenol	BNA	100 ug	100 ug

* At the time of injection.

- 4.2 Surrogate spike recovery must be evaluated by determining whether the concentration (measured as percent recovery) falls inside the contract required recovery limits listed in Table 4.2.

TABLE 4.2. CONTRACT REQUIRED SURROGATE SPIKE RECOVERY LIMITS

Fraction	Surrogate Compound	Water	Low/Medium Soil
BNA	Nitrobenzene-d ₅	35-114	23-120
BNA	2-Fluorobiphenyl	43-116	30-115
BNA	p-Terphenyl-d ₁₄	33-141	18-137
BNA	Phenol-d ₅	10-94	24-113
BNA	2-Fluorophenol	21-100	25-121
BNA	2,4,6-Tribromophenol	10-123	19-122

4.3 Treatment of surrogate spike recovery information is according to paragraphs 4.3.1 through 4.3.2.

4.3.1 Method Blank Surrogate Spike Recovery

The laboratory must take the actions listed below if recovery of any one surrogate compound in either the base/neutral or acid fraction is outside of contract surrogate spike recovery limits.

- 4.3.1.1** Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc; also check instrument performance.
- 4.3.1.2** Reanalyze the blank extract if steps in 4.3.1.1 fail to reveal the cause of the noncompliant surrogate recoveries.
- 4.3.1.3** Reextract and reanalyze the blank.
- 4.3.1.4** If the measures listed in 4.3.1.1 thru 4.3.1.3 fail to correct the problem, the analytical system must be considered to be out of control. The problem MUST be corrected before continuing. This may mean recalibrating the instrumentation but it may also mean more extensive action. The specific corrective action is left up to the GC/MS operator. When surrogate recovery(ies) in the blank is outside of the contract required windows, all samples associated with that blank MUST be reanalyzed at no additional cost to the Agency.

4.3.2 Sample Surrogate Spike Recovery

The laboratory must take the actions listed below if either of the following conditions exists:

- o** Recovery of any one surrogate compound in either base neutral or acid fraction is below 10%.
- o** Recoveries of two surrogate compounds in either base neutral or acid fractions are outside surrogate spike recovery limits.

- 4.3.2.1** The contractor shall document (in this instance, document means to write down and discuss the problem and corrective action taken in the Case Narrative, see Exhibit B) deviations outside of acceptable quality control limits and take the following actions:

4.3.2.1.1 Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc.; also check instrument performance.

4.3.2.1.2 If the steps in 4.3.2.1.1 fail to reveal a problem, then reanalyze the extract. If reanalysis of the extract solves the problem, then the problem was within the laboratory's control. Therefore, only submit data from the analysis with surrogate spike recoveries within the contract windows. This shall be considered the initial analysis and shall be reported as such on all data deliverables.

4.3.2.1.3 If the steps in 4.3.2.1.2 fail to solve the problem, then reextract and reanalyze the sample. If the reextraction and reanalysis solves the problem, then the problem was in the laboratory's control. Therefore, only submit data from the extraction and analysis with surrogate spike recoveries within the contract windows. This shall be considered the initial analysis and shall be reported as such on all data deliverables.

If the reextraction and reanalysis of the sample does not solve the problem; i.e., surrogate recoveries are outside the contract windows for both analyses, then submit the surrogate spike recovery data and the sample data from both analyses according to paragraph 4.4. Distinguish between the initial analysis and the reanalysis on all data deliverables, using the sample suffixes specified in Exhibit B.

4.4 Documentation

The contractor shall report surrogate recovery data for the following:

- o Method Blank Analysis
- o Sample Analysis
- o Matrix Spike/Matrix Spike Duplicate Analyses
- o All sample reanalyses that substantiate a matrix effect

The surrogate spike recovery data is summarized on the Surrogate Spike Percent Recovery Summary (Form II). Detailed instructions for the completion of Form II are in Exhibit B, Section III.

PART 5 - MATRIX SPIKE/MATRIX SPIKE DUPLICATE ANALYSIS (MS/MSD)**5. Summary**

In order to evaluate the matrix effect of the sample upon the analytical methodology, the USEPA has developed the standard mixes listed in Table 5.1 to be used for matrix spike and matrix spike duplicate analyses. These compounds are subject to change depending upon availability and suitability for use as matrix spikes.

5.1 MS/MSD Frequency of Analysis

A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix, once:

- o each Case of field samples received, OR
- o each 20 field samples in a Case, OR
- o each group of samples of a similar concentration level (soils only), OR
- o each 14 calendar day period during which samples in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group),

whichever is most frequent.

- 5.2 Use the compounds listed in Table 5.1 to prepare matrix spiking solutions according to protocols described in Exhibit D SV. The analytical protocols in Exhibit D SV stipulate the amount of matrix spiking solution to be added to the sample aliquots prior to extraction. Each method allows for optional dilution steps which must be accounted for when calculating percent recovery of the matrix spike and matrix spike duplicate samples.

TABLE 5.1. MATRIX SPIKING SOLUTIONS

<u>Base/Neutrals</u>	<u>Acids</u>
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-Methylphenol
N-Nitroso-Di-n-Propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

- 5.2.1 Samples requiring optional dilutions and chosen as the matrix spike/ matrix spike duplicate samples, must be analyzed at the same dilution as the original unspiked sample.

- 5.3 Individual component recoveries of the matrix spike are calculated using Equation 5.1.

$$\text{Matrix Spike Percent Recovery} = \frac{\text{SSR} - \text{SR}}{\text{SA}} \times 100 \quad \text{Eq. 5.1}$$

where

SSR = Spike Sample Results
 SR = Sample Result
 SA = Spike Added from spiking mix

- 5.4 Relative Percent Difference (RPD)

The contractor is required to calculate the relative percent difference between the matrix spike and matrix spike duplicate. The relative percent differences (RPD) for each component are calculated using Equation 5.2.

$$\text{RPD} = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100 \quad \text{Eq. 5.2}$$

where

RPD = Relative Percent Difference
 D₁ = First Sample Value
 D₂ = Second Sample Value (duplicate)

- 5.5 Documentation

The matrix spike (MS) results (concentrations) for nonspiked semi-volatile TCL compounds shall be reported on Form I (Organic Analysis Data Sheet) and the matrix spike percent recoveries shall be summarized on Form III (MS/MSD Recovery). These values will be used by EPA to periodically update existing performance based QC recovery limits (Table 5.2).

The results for nonspiked semivolatile TCL compounds in the matrix spike duplicate (MSD) analysis shall be reported on Form I (Organic Analysis Data Sheet) and the percent recovery and the relative percent difference shall be summarized on Form III (MS/MSD Recovery). The RPD data will be used by EPA to evaluate the long term precision of the analytical method. Detailed instructions for the completion of Form III are in Exhibit B, Section III.

TABLE 5.2. MATRIX SPIKE RECOVERY LIMITS

Fraction	Matrix Spike Compound	Water	Soil/Sediment
BN	1,2,4-Trichlorobenzene	39-98	38-107
BN	Acenaphthene	46-118	31-137
BN	2,4-Dinitrotoluene	24-96	28-89
BN	Pyrene	26-127	35-142
BN	N-Nitroso-Di-n-Propylamine	41-116	41-126
BN	1,4-Dichlorobenzene	36-97	28-104
Acid	Pentachlorophenol	9-103	17-109
Acid	Phenol	12-89	26-90
Acid	2-Chlorophenol	27-123	25-102
Acid	4-Chloro-3-Methylphenol	23-97	26-103
Acid	4-Nitrophenol	10-80	11-114

PART 6 - SAMPLE ANALYSIS

6. Summary

The intent of Part 6 is to provide the Contractor with a brief summary of ongoing QC activities involved with sample analysis. Specific references are provided to help the Contractor meet specific reporting and deliverables requirements of this contract.

6.1 Sample Analysis

Samples can be analyzed upon successful completion of the initial QC activities. When twelve (12) hours have elapsed since the initial tune was completed, it is necessary to conduct an instrument tune and calibration check analysis (described in Part 2 of this Section). Any major system maintenance, such as a source cleaning or installation of a new column, may necessitate a retune and recalibration (see Initial Calibration, Part 2). Minor maintenance should necessitate only the calibration verification (Continuing Calibration, Part 2).

6.1.1 Internal Standards Evaluation - Internal standard responses and retention times in all samples must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate. The criteria are described in detail in the instructions for Form VIII, Internal Standard Area Summary (see Exhibit B, Section III). If the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to

100%), from the latest daily (12 hour time period) calibration standard, the mass spectrometric system must be inspected for malfunction, and corrections made as appropriate. Breaking off 1 foot of the column or cleaning the injector sleeve will often improve high end sensitivity for the late eluting compounds; repositioning or repacking the front end of the column will often improve front end column performance. Poor injection technique can also lead to variable IS ratios. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

6.1.1.1 If after reanalysis, the EICP areas for all internal standards are inside the contract limits (-50% to +100%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, only submit data from the analysis with EICP's within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

6.1.1.2 If the reanalysis of the sample does not solve the problem, i.e., the EICP areas are outside contract limits for both analyses, then submit the EICP data and sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables, using the sample suffixes specified in Exhibit B. Document in the Case Narrative all inspection and corrective actions taken.

6.1.2 Each analytical run must also be checked for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound. The initial method calibration (Part 2) requires that the system should not be saturated for high response compounds at 160 nanograms for semivolatile TCL compounds.

6.1.2.1 If any compound in any sample exceeds the initial calibration range, that sample must be diluted, the internal standard concentration readjusted, and the sample reinjected, as described in specific methodologies in Exhibit D SV. Secondary ion quantitation is only allowed when there are sample matrix interferences with the primary ion.

6.1.2.2 If the dilution of the sample causes any compound detected in the first analysis to be undetectable in the second analysis, then the results of both analyses shall be reported on separate Forms I, according to the instructions in Exhibit B.

6.1.3 Qualitative Analysis

The semivolatile compounds listed in the Target Compound List (TCL), Exhibit C, shall be identified by an analyst competent in the interpretation of mass spectra, by comparison of the suspect mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications: (1) elution of the sample component at the same GC relative retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra (see Exhibit D SV, Section IV).

6.1.3.1 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample.

6.1.3.2 For comparison of standard and sample component mass spectra, mass spectra obtained on the Contractor's GC/MS are required. The DFTPP tuning requirements listed in Part I must be met on the same GC/MS.

6.1.3.2.1 The requirements for qualitative verification by comparison of mass spectra are as follows:

- o All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.
- o The relative intensities of ions specified in the above paragraph must agree within $\pm 20\%$ between the standard and sample spectra.
- o Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. When GC/MS computer data processing programs are used to obtain the sample component spectrum, both the processed and the raw spectra must be evaluated. In Task III, the verification process should favor false positives (Exhibit D SV, Section IV).

6.1.3.2.2 If a compound cannot be verified by all of the criteria in 6.1.3.2.1, but in the technical judgement of the mass spectral interpretation specialist the identification is correct, the contractor shall report the identification and proceed with the quantitation.

6.1.3.3 A library search shall be executed for non-surrogate and non-TCL sample components for the purpose of tentative identification. For this purpose, the 1985 or most recent available version of the National Bureau of Standards Mass Spectral Library, containing 42,261 spectra, should be used.

6.1.4 Quantitation

6.1.4.1 Semivolatile TCL components identified shall be quantitated by the internal standard method. The internal standards used shall be the ones assigned in Table 2.2 of this Section. The EICP area of characteristic ions of TCL analytes are used (Exhibit D SV, Section IV).

6.1.4.2 An estimated concentration for non-TCL components tentatively identified shall be quantitated by the internal standard method. For quantification, the nearest internal standard free of interferences must be used.

6.1.4.3 Calculate surrogate standard recovery (see Part 4) for all surrogate compounds on all samples, blanks, matrix spikes, and matrix spike duplicates. If recovery is within contractual limits, report on Form II (see Exhibit B, Section III). If recovery is outside contractual limits, take specific steps listed in Surrogate Spike Recoveries (Part 4).

6.1.4.4 Calculate matrix spike and matrix spike duplicate percent recovery (see Part 5) for all compounds and report results on Form III (see Exhibit B, Section III). Calculate Relative Percent Differences (RPD's) for all matrix spiking compounds and report results on Form III. Ensure that the proper frequency of MS/MSD analysis is maintained.

6.1.5 Reporting and Deliverables

Refer to Exhibit B of this Statement of Work for specific details on contract deliverables and reporting formats. Exhibit B contains specific instructions for completing all required Forms, as well as a detailed itemization of reporting and deliverables requirements. Exhibit H contains the format requirements for delivery of data in computer-readable format.

Section 5
Standard Operating Procedure
Total Phenolics

PHENOLICS, TOTAL RECOVERABLE

Method 420.1 (Spectrophotometric, Manual 4-AAP with Distillation)

STORET NO. 32730

1. Scope and Application
 - 1.1 This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes.
 - 1.2 The method is capable of measuring phenolic materials at the 5 ug/l level when the colored end product is extracted and concentrated in a solvent phase using phenol as a standard.
 - 1.3 The method is capable of measuring phenolic materials that contain more than 50 ug/l in the aqueous phase (without solvent extraction) using phenol as a standard.
 - 1.4 It is not possible to use this method to differentiate between different kinds of phenols.
2. Summary of Method
 - 2.1 Phenolic materials react with 4-aminoantipyrine in the presence of potassium ferricyanide at a pH of 10 to form a stable reddish-brown colored antipyrine dye. The amount of color produced is a function of the concentration of phenolic material.
3. Comments
 - 3.1 For most samples a preliminary distillation is required to remove interfering materials.
 - 3.2 Color response of phenolic materials with 4-amino antipyrine is not the same for all compounds. Because phenolic type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.
4. Sample Handling and Preservation
 - 4.1 Biological degradation is inhibited by the addition of 1 g/l of copper sulfate to the sample and acidification to a pH of less than 4 with phosphoric acid. The sample should be kept at 4°C and analyzed within 24 hours after collection.
5. Interference
 - 5.1 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of less than 4 with H_3PO_4 and aerating briefly by stirring and adding $CuSO_4$.
 - 5.2 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate (7.10). If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

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6. Apparatus

- 6.1 Distillation apparatus, all glass consisting of a 1 liter pyrex distilling apparatus with Graham condenser.
- 6.2 pH meter.
- 6.3 Spectrophotometer, for use at 460 or 510 nm.
- 6.4 Funnels.
- 6.5 Filter paper.
- 6.6 Membrane filters.
- 6.7 Separatory funnels, 500 or 1,000 ml.
- 6.8 Nessler tubes, short or long form.

7. Reagents

- 7.1 Phosphoric acid solution, 1 + 9: Dilute 10 ml of 85% H_3PO_4 to 100 ml with distilled water.
- 7.2 Copper sulfate solution: Dissolve 100 g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in distilled water and dilute to 1 liter.
- 7.3 Buffer solution: Dissolve 16.9 g NH_4Cl in 143 ml conc. NH_4OH and dilute to 250 ml with distilled water. Two ml should adjust 100 ml of distillate to pH 10.
- 7.4 Aminoantipyrine solution: Dissolve 2 g of 4AAP in distilled water and dilute to 100 ml.
- 7.5 Potassium ferricyanide solution: Dissolve 8 g of $\text{K}_3\text{Fe}(\text{CN})_6$ in distilled water and dilute to 100 ml.
- 7.6 Stock phenol solution: Dissolve 1.0 g phenol in freshly boiled and cooled distilled water and dilute to 1 liter. 1 ml = 1 mg phenol.
- 7.7 Working solution A: Dilute 10 ml stock phenol solution to 1 liter with distilled water. 1 ml = 10 μg phenol.
- 7.8 Working solution B: Dilute 100 ml of working solution A to 1000 ml with distilled water. 1 ml = 1 μg phenol.
- 7.9 Chloroform
- 7.10 Ferrous ammonium sulfate: Dissolve 1.1 g ferrous ammonium sulfate in 500 ml distilled water containing 1 ml conc. H_2SO_4 and dilute to 1 liter with freshly boiled and cooled distilled water.

8. Procedure

8.1 Distillation

- 8.1.1 Measure 500 ml sample into a beaker. Lower the pH to approximately 4 with 1 + 9 H_3PO_4 (7.1), add 5 ml CuSO_4 solution (7.2) and transfer to the distillation apparatus. Omit adding H_2PO_4 and CuSO_4 if sample was preserved as described in 4.1.
- 8.1.2 Distill 450 ml of sample, stop the distillation, and when boiling ceases add 50 ml of warm distilled water to the flask and resume distillation until 500 ml have been collected.
- 8.1.3 If the distillate is turbid, filter through a prewashed membrane filter.

8.2 Direct photometric method

- 8.2.1 Using working solution A (7.7), prepare the following standards in 100 ml volumetric flasks.

<u>ml of working solution A</u>	<u>Conc. ug/l</u>
0	0.0
0.5	50.0
1.0	100.0
2.0	200.0
5.0	500.0
8.0	800.0
10.0	1000.0

8.2.2 To 100 ml of distillate or an aliquot diluted to 100 ml and/or standards, add 2 ml of buffer solution (7.3) and mix. The pH of the sample and standards should be 10 ± 0.2 .

8.2.3 Add 2.0 ml aminoantipyrine solution (7.4) and mix.

8.2.4 Add 2.0 ml potassium ferricyanide solution (7.5) and mix.

8.2.5 After 15 minutes read absorbance at 510 nm.

8.3 Chloroform extraction method

8.3.1 Using working solution B (7.8), prepare the following standards. Standards may be prepared by pipetting the required volumes into the separatory funnels and diluting to 500 ml with distilled water.

<u>ml of working solution B</u>	<u>Conc. ug/l</u>
0.0	0.0
3.0	6.0
5.0	10.0
10.0	20.0
20.0	40.0
25.0	50.0

8.3.2 Place 500 ml of distillate or an aliquot diluted to 500 ml in a separatory funnel. The sample should not contain more than 25 ug phenol.

8.3.3 To sample and standards add 10 ml of buffer solution (7.3) and mix. The pH should be 10 ± 0.2 .

8.3.4 Add 3.0 ml aminoantipyrine solution (7.4) and mix.

8.3.5 Add 3.0 ml potassium ferricyanide solution (7.5) and mix.

8.3.6 After three minutes, extract with 25 ml of chloroform (7.9). Shake the separatory funnel at least 10 times, let CHCl_3 settle, shake again 10 times and let chloroform settle again. Vent chloroform fumes into hood.

8.3.7 Filter chloroform extracts through filter paper. Do not add more chloroform. Carryout filtration in a hood. Dispose of chloroform in environmentally acceptable manner.

8.3.8 Read the absorbance of the samples and standards against the blank at 460 nm.

9. Calculation

9.1 Prepare a standard curve by plotting the absorbance value of standards versus the corresponding phenol concentrations.

9.2 Obtain concentration value of sample directly from standard curve.

10. Precision and Accuracy

10.1 Using the extraction procedure for concentration of color, six laboratories analyzed samples at concentrations of 9.6, 48.3, and 93.5 ug/l. Standard deviations were ± 0.99 , ± 3.1 and ± 4.2 ug/l, respectively.

10.2 Using the direct photometric procedure, six laboratories analyzed samples at concentrations of 4.7, 48.2 and 97.0 mg/l. Standard deviations were ± 0.18 , ± 0.48 and ± 1.58 mg/l, respectively.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D1783-70, p553 (1976).
2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p574-581, Method 510 through 510C, (1975).

PHENOLICS, TOTAL RECOVERABLE

Method 420.2 (Colorimetric, Automated 4-AAP with Distillation)

STORET NO. 32730

1. Scope and Application
 - 1.1 This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes.
 - 1.2 The method is capable of measuring phenolic materials from 2 to 500 $\mu\text{g}/\text{l}$ in the aqueous phase using phenol as a standard. The working ranges are 2 to 200 $\mu\text{g}/\text{l}$ and 10 to 500 $\mu\text{g}/\text{l}$.
2. Summary of Method
 - 2.1 This automated method is based on the distillation of phenol and subsequent reaction of the distillate with alkaline ferricyanide and 4-aminoantipyrine to form a red complex which is measured at 505 or 520 nm. The same manifold is used with the AAI or AAI.
3. Sample Handling and Preservation
 - 3.1 Biological degradation is inhibited by the addition of 1 g/l of copper sulfate to the sample and acidification to a pH of less than 4 with phosphoric acid. The sample should be kept at 4°C and analyzed within 24 hours after collection.
4. Interference
 - 4.1 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of less than 4.0 with H_3PO_4 and aerating briefly by stirring and adding CuSO_4 .
 - 4.2 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate (6.5). If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.
 - 4.3 Background contamination from plastic tubing and sample containers is eliminated by filling the wash receptacle by siphon (using Kel-F tubing) and using glass tubes for the samples and standards.
5. Apparatus
 - 5.1 Technicon AutoAnalyzer (I or II)
 - 5.1.1 Sampler equipped with continuous mixer.
 - 5.1.2 Manifold.
 - 5.1.3 Proportioning pump II or III.
 - 5.1.4 Heating bath with distillation coil.
 - 5.1.5 Distillation head.
 - 5.1.6 Colorimeter equipped with a 50 mm flow cell and 505 or 520 nm filter.
 - 5.1.7 Recorder.
6. Reagents
 - 6.1 Distillation reagent: Add 100 ml of conc. phosphoric acid (85% H_3PO_4) to 800 ml of distilled water, cool and dilute to 1 liter.

Issued 1974

- 6.2 Buffered potassium ferricyanide: Dissolve 2.0 g potassium ferricyanide, 3.1 g boric acid and 3.75 g potassium chloride in 800 ml of distilled water. Adjust to pH of 10.3 with 1 N sodium hydroxide (6.3) and dilute to 1 liter. Add 0.5 ml of Brij-35. Prepare fresh weekly.
- 6.3 Sodium hydroxide (1N): Dissolve 40 g NaOH in 500 ml of distilled water, cool and dilute to 1 liter.
- 6.4 4-Aminoantipyrine: Dissolve 0.65 g of 4-aminoantipyrine in 800 ml of distilled water and dilute to 1 liter. Prepare fresh each day.
- 6.5 Ferrous ammonium sulfate: Dissolve 1.1 g ferrous ammonium sulfate in 500 ml distilled water containing 1 ml H_2SO_4 and dilute to 1 liter with freshly boiled and cooled distilled water.
- 6.6 Stock phenol: Dissolve 1.00 g phenol in 500 ml of distilled water and dilute to 1000 ml. Add 1 g CuSO_4 and 0.5 ml conc. H_3PO_4 as preservative. 1.0 ml = 1.0 mg phenol.
- 6.7 Standard phenol solution A: Dilute 10.0 ml of stock phenol solution (6.6) to 1000 ml. 1.0 ml = 0.01 mg phenol.
- 6.8 Standard phenol solution B: Dilute 100.0 ml of standard phenol solution A (6.7) to 1000 ml with distilled water. 1.0 ml = 0.001 mg phenol.
- 6.9 Standard solution C: Dilute 100.0 ml of standard phenol solution B (6.8) to 1000 ml with distilled water. 1.0 ml = 0.0001 mg phenol.
- 6.10 Using standard solution A, B or C prepare the following standards in 100 ml volumetric flasks. Each standard should be preserved by adding 0.1 g CuSO_4 and 2 drops of conc. H_3PO_4 to 100.0 ml.

<u>ml of Standard Solution Solution C</u>	<u>Conc. ug/l</u>
1.0	1.0
2.0	2.0
3.0	3.0
5.0	5.0
<u>Solution B</u>	
1.0	10.0
2.0	20.0
5.0	50.0
10.0	100.0
<u>Solution A</u>	
2	200
3	300
5	500

7. Procedure

- 7.1 Set up the manifold as shown in Figures 1 or 2.
- 7.2 Fill the wash receptacle by siphon. Use Kel-F tubing with a fast flow (1 liter/hr).
- 7.3 Allow colorimeter and recorder to warm up for 30 minutes. Run a baseline with all reagents, feeding distilled water through the sample line. Use polyethylene tubing for

sample line. When new tubing is used, about 2 hours may be required to obtain a stable baseline. This two hour time period may be necessary to remove the residual phenol from the tubing.

- 7.4 Place appropriate phenol standards in sampler in order of decreasing concentration. Complete loading of sampler tray with unknown samples, using glass tubes.

NOTE 1: If samples have not been preserved as instructed in (3.1), add 0.1 g CuSO_4 and 2 drops of conc. H_3PO_4 to 100 ml of sample.

- 7.5 Switch sample line from distilled water to sampler and begin analysis.

8. Calculation

- 8.1 Prepare standard curve by plotting peak heights of standards against concentration values. Compute concentration of samples by comparing sample peak heights with standards.

9. Precision and Accuracy

- 9.1 In a single laboratory (EMSL), using sewage samples at concentrations of 3.8, 15, 43 and 89 $\mu\text{g}/\text{l}$, the standard deviations were ± 0.5 , ± 0.6 , ± 0.6 and $\pm 1.0 \mu\text{g}/\text{l}$, respectively. At concentrations of 73, 146, 299 and 447 $\mu\text{g}/\text{l}$, the standard deviations were ± 1.0 , ± 1.8 , ± 4.2 and $\pm 5.3 \mu\text{g}/\text{l}$, respectively.
- 9.2 In a single laboratory (EMSL), using sewage samples at concentrations of 5.3 and 82 $\mu\text{g}/\text{l}$, the recoveries were 78% and 98%. At concentrations of 168 and 489 $\mu\text{g}/\text{l}$, the recoveries were 97% and 98%, respectively.

Bibliography

1. Technicon AutoAnalyzer II Methodology, Industrial Method No. 127-71W, AAII.
2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 574, Method 510 (1975).
3. Gales, M.E. and Booth, R.L., "Automated 4 AAP Phenolic Method", AWWA 68, 540 (1976).

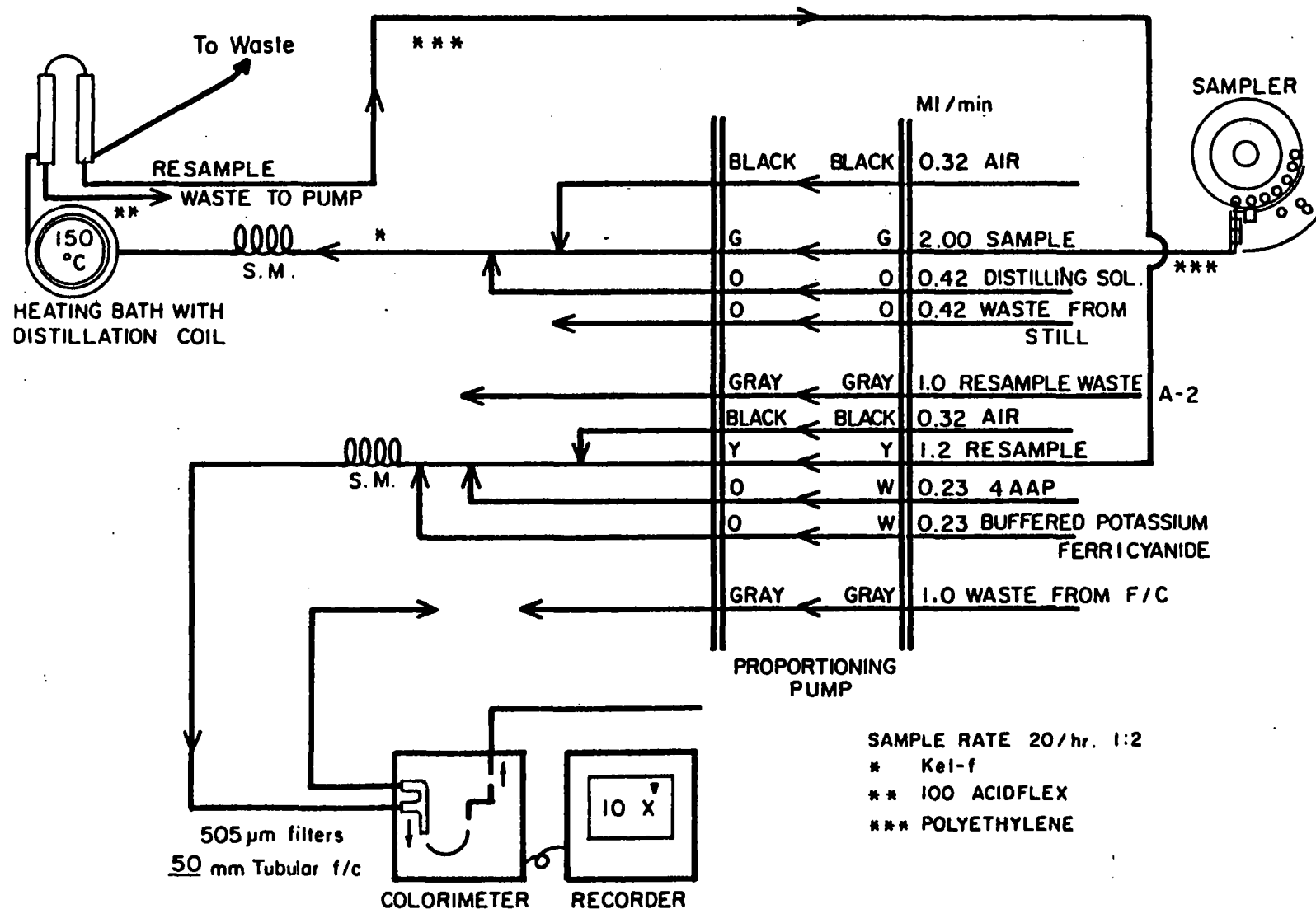


FIGURE 1. PHENOL AUTO ANALYZER I

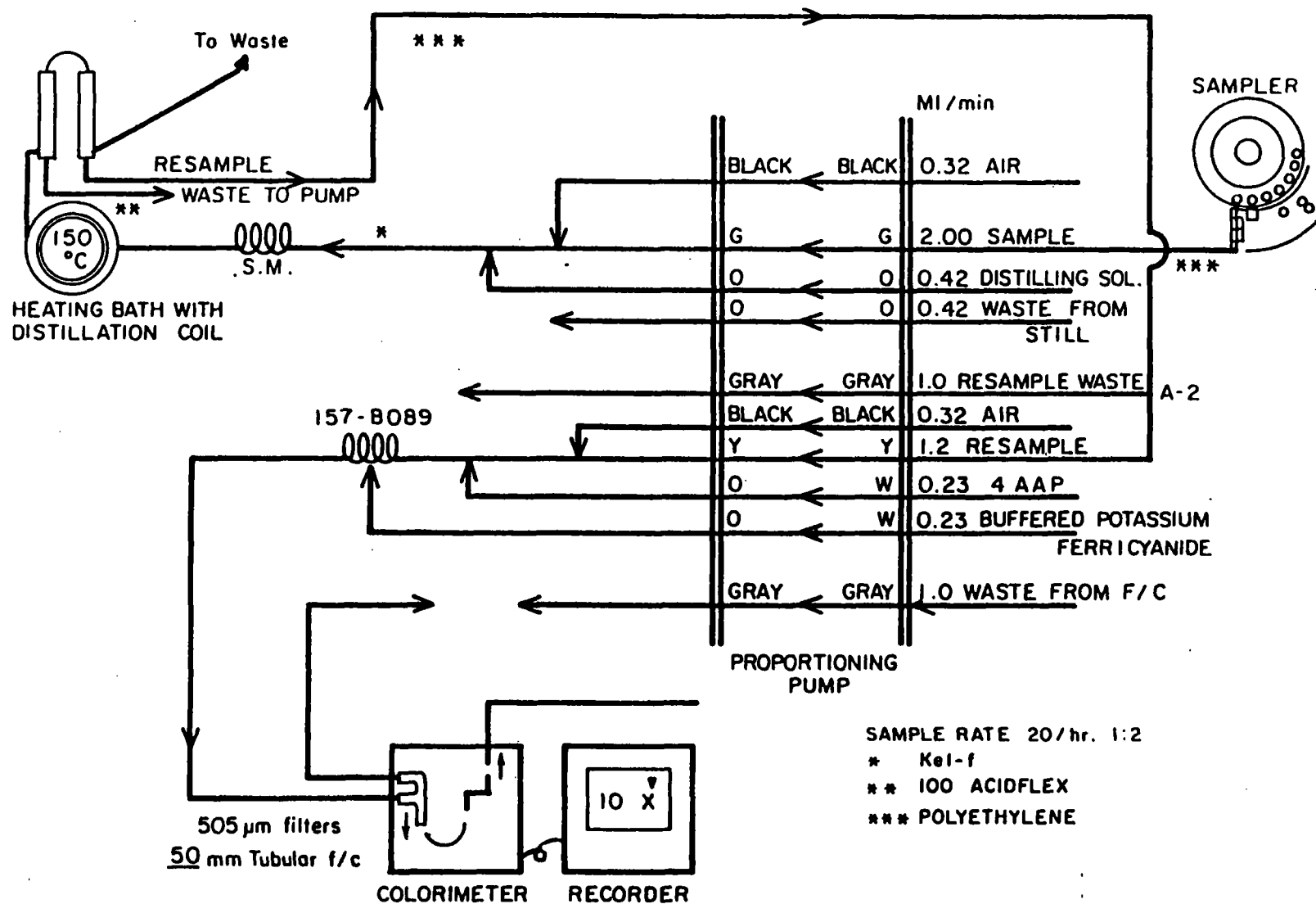


FIGURE 2. PHENOL AUTO ANALYZER II

The following background and history of response actions is an excerpt from the MPCA Board Item on April 22, 1986 "Request for Approval of a Consent Decree with the Reilly Tar & Chemical Corporation and Other Parties for the Purpose of Completing Remedial Investigations and Feasibility Studies and Developing and Implementing Response Actions at and Around the Reilly Tar Site in St. Louis Park". This material is in Section I of the Issue Statement of that MPCA Board Item.

Between 1917 and 1972, Reilly Tar & Chemical Corporation (Reilly) operated a coal tar distillation and wood preserving plant, known as the Republic Creosote Company, in St. Louis Park. This plant was located on an 80 acre tract near State Highway 7 and Louisiana Avenue (the Reilly Site; Figure 1). Reilly disposed of wastewater from the operation in a network of ditches which discharged into a swamp south of the Reilly Site. In addition, the wood treating activities conducted on the Reilly Site resulted in creosote and coal tar contamination of the soils from drippings and spills. The major constituents of coal tar are phenolic compounds and polynuclear aromatic hydrocarbons (PAH). Some PAH compounds are carcinogenic, and are thus a source of concern when a municipal drinking water supply is contaminated with these compounds. (As used in the remainder of this board item, "contaminated" or "contamination" means PAH or phenolics are present in soil or ground water resulting from activities of Reilly at the Reilly Site.)

QUALITY ASSURANCE BRANCH

APR 05 1988

ENVIRONMENT SERVICES DIVISION

In 1932, the first municipal well in St. Louis Park (Old SLP #1) was constructed at Brunswick Avenue and West 36th Street, approximately one-half mile east of the Reilly Site. The well was finished in the Prairie du Chien-Jordan aquifer (Figure 2). After several weeks of operation, the well was closed due to taste and odor complaints (the taste was described as "swampy"). Laboratory tests showed that phenolic compounds were the apparent cause of the problem. Phenolics cause water to have an unpleasant taste and odor when the water is chlorinated, but these compounds are not believed to have adverse health effects at the low levels which cause the taste and odor problems.

Attempts to remedy the situation were unsuccessful, and the well was abandoned. Well drillers at the time speculated that the Reilly Site might be the source of the problem. Although Reilly, at the time, insisted that the problems at the municipal well were the result of "decaying vegetation" from the swamp south of the Reilly Site, it filled an unused well (W105) located on the Reilly Site with sand and extended the casings in Reilly's main water supply well (W23) to reduce interaquifer flow of possibly contaminated water.

Complaints from nearby residences over contamination of shallow wells and of odors from air emissions became more common, especially after extensive residential development of the area during the late 1940's into the 1950's. Because of continuing problems with soil and surface water contamination and odors 1/, the City of St. Louis Park (City) and the Minnesota Pollution Control Agency (MPCA) through the Attorney General (the State) filed suit against Reilly in 1970. In 1972, the City purchased the Reilly Site from Reilly, and

1/ See paragraph thirteen, page 9, of the attached Consent Decree for a listing of various studies and/or reports, chemical analysis and field investigations relating to the Reilly Site.

the plant was dismantled and removed. The City intended to use the property for a realignment of Louisiana Avenue and for residential development, and dropped its lawsuit against Reilly as a condition of the sale. However, the State did not drop the lawsuit, which is still pending and will be dismissed as part of the proposed settlement.

In 1974, the City contracted with Gerald Sunde, a consulting engineer, to investigate pathways for the movement of contaminants. Sunde concluded that wells in the area open to several aquifers (multi-aquifer wells) provide a significant pathway for the spread of contamination from contaminated surficial aquifers to deeper aquifers which would otherwise be protected from contamination by several bedrock layers. In 1975, the MPCA contracted with Barr Engineering to investigate subsurface contamination at and south of the Reilly Site. The results of this study showed significant contamination of soil and the surficial aquifer (the drift) with creosote. Because it appeared that Sunde's assessment of the pathways for contamination to deep aquifers was, at least in part, correct, the Minnesota Department of Health (MDH) in 1978 and 1979 contracted for the closure of 29 multi-aquifer wells in areas where the surficial aquifers were the most contaminated. In addition, the City and the U.S. Geological Survey installed a packer and casing in the former Reilly well, W23, to stop the extensive downhole flow of contaminated water into the Prairie du Chien - Jordan Aquifer.

Louisiana Avenue was constructed through the Reilly Site during the mid-1970's, and some multi-family housing units were constructed in the northern half of the Reilly Site during this same time period.

In 1978 the MDH began analyses of water from municipal supply wells in St. Louis Park and neighboring communities for PAH using high performance liquid chromatography. These and subsequent analyses led to the discovery of significant concentrations of PAH in six St. Louis Park wells and one Hopkins well, and these wells were shut down during the period 1978-81.

As a result of the determination that area ground water was contaminated the State amended, in 1978, its complaint in the lawsuit with Reilly to include claims for ground water contamination. All of the municipal wells cited above are finished in the Prairie du Chien-Jordan aquifer, which is the most heavily used aquifer for municipal drinking water supplies in the Twin Cities metropolitan area. The City of St. Louis Park has since overcome part of the resulting water supply shortfall through water conservation measures, installation of a new well in the Mt. Simon-Hinckley aquifer, and an interconnection with the City of Plymouth. In an attempt to understand the processes of contaminant transport in the Prairie du Chien-Jordan, the MDH and MPCA contributed toward a ground water flow and contaminant transport modeling study performed by the United States Geological Survey (USGS). In addition, the MDH funded a study by Hickok and Associates of the feasibility of ground water gradient control 2/ in 1981.

The MPCA received a \$400,000 grant from the U.S. Environmental Protection Agency (EPA) in December, 1981, and used this grant to finance a feasibility study conducted by the MPCA contractor, CH2M Hill, for replacement or treatment

2/ The term "gradient control", as used in this discussion, refers to the utilization of a pumping well or wells, usually located near the leading edge of the contamination plume, to control the flow of ground water in an aquifer to contain contamination within the area of control. It is in contrast to "source control", in which highly contaminated water is pumped at or near the source.

of the lost water supply; and to locate, investigate, and close multi-aquifer wells. In December, 1982, the EPA awarded the MPCA a \$1.99 million grant under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) to continue these studies, and to provide more funding for the USGS ground water modeling study for the purpose of designing a gradient control well system to control the movement of contaminants in the Prairie du Chien-Jordan aquifer.

The results of these studies have provided sufficient information to design a remedial approach for the contamination in the Prairie du Chien-Jordan aquifer. Multi-aquifer well investigation under the CERCLA grant has been limited to date, to two priority wells located on the Reilly Site. The most important of these, W23, was found to have a plug of coal tar in it, and has been cleaned out. The other well, W105, was the water supply well for the sugar beet plant which occupied the Reilly Site around the turn of the century, and was used by Reilly as a backup supply well until 1933. W105 was not found to be a source of contamination as was W23. The drinking water restoration study conducted by CH2M Hill, which analyzed the feasibility of various methods of treating drinking water, deeper wells, and an interconnection with Minneapolis, concluded that treatment with granular activated carbon (GAC) was the most cost-effective method of restoring the City's lost water supply. The USGS has completed its ground water flow modeling work, and this model has been used to examine gradient control schemes.

In September, 1980, the EPA filed suit against Reilly alleging violation of the Resource Conservation Recovery Act (RCRA). The State and St. Louis Park joined the lawsuit in October, 1980, followed by the City of Hopkins in June, 1981. After passage of CERCLA, the State filed an amended complaint in May, 1981, followed by St. Louis Park, the EPA, and Hopkins respectively in

August, September, and October, 1981. The State filed a second amended complaint under the Minnesota Environmental Response and Liability Act ("MERLA") in 1985 followed by St. Louis Park and Hopkins. In the event that agreement is not reached on the Consent Decree, the case is scheduled to be heard before U.S. District Court Judge Paul Magnuson.

In addition to the above litigation, Reilly filed a counterclaim against St. Louis Park, and St. Louis Park asserted a cross-claim against the State. Other parties involved with the purchase and development of the northern portion of the Reilly Site filed cross-claims against St. Louis Park and Reilly. 3/

In May, 1983, Reilly and its consultant, Environmental Research & Technology, Inc. (ERT) issued a report on the St. Louis Park ground water contamination. Discussion among MPCA, EPA, Reilly and ERT staff led to a period of negotiations toward a settlement. These talks broke down in early 1984.

Both the MPCA and EPA have instituted administrative action against Reilly, pursuant to the respective State and federal Superfund acts, in order to compel Reilly to undertake necessary remedial actions. The EPA issued a Record of Decision (ROD) in June, 1984 affirming that the most cost-effective remedy for restoring the City's lost water supply was installation of a GAC treatment system. In August, 1984, the EPA issued to Reilly an Administrative Order directing Reilly to design and construct the GAC system for City wells SLP 10 and 15 as provided in the ROD. In December, 1984, the MPCA issued a Request for Response Action (RFRA) to Reilly outlining a range of remedial investigations, feasibility studies, and necessary remedial actions.

3/ The following organizations were included as defendants in the lawsuit because they were involved with the purchase and development of the northern portion of the Reilly Site from St. Louis Park: Housing and Redevelopment Authority of St. Louis Park, Oak Park Village Associates, and Philips Investment Co.

Following these administrative actions, extensive negotiations, which had previously broken down, resumed among the MPCA, EPA, St. Louis Park, and Reilly in an effort to reach an effective settlement. General agreement on the terms and conditions of a proposed Consent Decree was reached in the Summer of 1985. However, because of its complex nature and the large number of parties involved, final agreement was delayed. Since the likelihood of settlement was always present, the MPCA staff did not return to the MPCA Board for further administrative actions.

Since general agreement regarding a Consent Decree had been reached in mid 1985, Reilly did proceed to design and construct a GAC system for City wells SLP 10 and 15. Reilly completed the construction of the GAC system in December, 1985 and the system is expected to be operational by May, 1986.

The following background and history of response actions is Part C of the Consent Decree. A list of relevant reference documents is included.

1. From 1917 until 1972, Reilly was engaged in the business of coal tar distillation and pressure treatment of wood products at its plant site at 7200 Walker Street, St. Louis Park, Hennepin County, Minnesota (hereinafter "the Site"). The Site encompassed an eighty (80) acre tract, which consists of Lot 1, Block 1; Lot 1, Block 2; Lot 1, Block 3; Lot 1, Block 4; Lot 1, Block 5; Lot 1, Block 6; Lot 1, Block 7; Lot 1, Block 8; Lot 1, Block 9; Lot 1, Block 10; all in Oak Park

Village according to the plat thereof on file in the office of the County Recorder of Hennepin County, Minnesota.

2. On or about October 2, 1970, the State, through its Pollution Control Agency, and St. Louis Park, filed a complaint in the Hennepin County District Court of the State of Minnesota alleging violations by Reilly of state and municipal pollution control laws and regulations. State of Minnesota by the Minnesota Pollution Control Agency, and the City of St. Louis Park v. Reilly Tar & Chemical Corporation, Hennepin County District Court, Civil File No. 670767 (hereinafter "Hennepin County Lawsuit").

3. On April 14, 1972, St. Louis Park agreed to purchase the Site from Reilly. The purchase agreement included a promise by St. Louis Park to obtain dismissals with prejudice by the State and by St. Louis Park of the Hennepin County Lawsuit. The purchase agreement also provided for acceptance by St. Louis Park of the property in an "as is" condition, including "any and all questions of soil and water impurities and soil conditions," and an agreement by St. Louis Park "to make no claim against Reilly for damages relative to soil and water impurities, if any, in any way relating to the premises sold herein, or relative to any other premises in which the City of St. Louis Park holds an interest. . . ."

4. A closing was scheduled on the property for June 19, 1973. However, the State did not execute a dismissal

of the Hennepin County Lawsuit. Accordingly, the City of St. Louis Park agreed that it would "hold Reilly harmless from any and all claims which may be asserted against it by the State of Minnesota, acting by and through the Minnesota Pollution Control Agency, and will be fully responsible for restoring the property, at its expense, to any condition that may be required by the Minnesota Pollution Control Agency". The City of St. Louis Park and Reilly executed and filed dismissals with prejudice of their claims in the Hennepin County Lawsuit, and the closing took place thereafter.

5. On June 21, 1973, the property was conveyed by quitclaim deed from St. Louis Park to the Housing and Redevelopment Authority of St. Louis Park, Minnesota, which thereafter conveyed part of the property to Oak Park Village Associates, Rustic Oaks Condominium, Inc. and Philip's Investment Co.

The Agreement for Purchase and Sale of Real Estate dated October 4, 1977 and the First Addendum to the Agreement dated October 6, 1977 between the St. Louis Park Housing and Redevelopment Authority and Diversified Equities Corporation [Oak Park Village Associates] regarding Lot 1, Block 3, Oak Park Village, Hennepin County, Minnesota, provides as follows:

14. Environmental Matters

The Agency [St. Louis Park Housing and Redevelopment Authority] shall prepare and shall incur all expenses for any environmental approvals, assessments, environmental impact

statements or such other environmental review documents deemed necessary or desirable by governmental authority.

Agency [St. Louis Park Housing and Redevelopment Authority] agrees to indemnify and save Redeveloper harmless from and against any and all loss or damage Redeveloper or successors may suffer from damage to improvements constructed on the Property as a result of claims, demands, costs or judgments against and arising out of soil or ground water contamination existing as of the date hereof, or caused by conditions existing as of the date hereof.

The Agreement for Purchase and Sale of Real Estate dated June 1, 1979 by and between the Housing and Redevelopment Authority of St. Louis Park and Ben Weber [Philip's Investment Co.] and the City of St. Louis Park regarding Lot 1, Block 6, Oak Park Village, Hennepin County, Minnesota, provides as follows:

14. Environmental Matters.

a. Both the City and the Redeveloper agree that the Stipulation between the City and the PCA dated April 19, 1977, is capable of a possible variety of interpretations. As between the Agency [St. Louis Park Housing and Redevelopment Authority], the City and the Redeveloper, as an inducement to the City and Agency to allow the Redeveloper to develop the Property and as security against the Redeveloper, or its assigns or successors in interest, claiming the right to benefit from a broader interpretation of said Stipulation and as an inducement to the Redeveloper to develop the Property and as security against the City or Agency claiming the right to benefit from a narrower interpretation of said Stipulation, the City, Agency and Redeveloper agree that, as between the parties to this Agreement, this paragraph 14 shall constitute the sole remedy available to Redeveloper against the City and Agency for any action or claim against or loss or damage to the

Redeveloper which is based on, derived from, or related to the soil or groundwater conditions of the Property, and shall constitute, as between the parties to this agreement, their interpretation of the Stipulation.

b. The City will not require the Redeveloper to excavate soil from the Property in question because of soil or groundwater contamination resulting from the operations of the former Republic Creosote Plant.

c. The City will indemnify the Redeveloper from damage consisting of physical destruction or injury to improvements on the property due solely to soil excavation on the Property required by public agencies. This indemnification shall not include consequential damage, lost income, lost profit or other forms of indirect loss or damage nor shall it include damage arising from personal injury. Indemnification shall be on a replacement cost less depreciation basis.

d. The indemnification granted by this agreement shall be secondary to any other rights or potential rights which the Redeveloper may have to compensation for any damage or loss whether through eminent domain, grants or otherwise. The Redeveloper shall exercise good faith effort to seek and obtain such compensation before presenting a claim under this indemnification agreement. Any compensation from any other source for damages indemnified herein shall reduce the indemnification liability of the City dollar per dollar.

e. This indemnification and agreement shall not be assignable except to the first mortgagee and shall terminate on January 1, 1985. All claims to indemnification under this agreement must be made in writing and received by the City Clerk of the City prior to January 2, 1985.

6. In April, 1978, the State moved to amend its complaint in the Hennepin County Lawsuit, alleging that PAH substances contained in Reilly's coal tar and creosote wastes had entered the ground water beneath the Site and that their

further migration threatened to contaminate aquifers relied on for public water supply. At the same time, St. Louis Park moved to intervene as a plaintiff. The motions were granted and interlocutory review was denied by the Minnesota Supreme Court. Reilly subsequently tendered defense of the action to St. Louis Park and counterclaimed against St. Louis Park, asserting that St. Louis Park was responsible for dealing with this problem under the hold harmless agreement made at the time of its purchase of the Site.

7. On or about September 4, 1980, the United States commenced this action by filing a complaint under Section 7003 of the Resource Conservation and Recovery Act ("RCRA"), 42 U.S.C. § 6973, alleging, inter alia, the existence of an imminent and substantial endangerment to health and the environment due to the handling, treatment, storage, transportation, disposal and presence of hazardous waste at the Site. On or about October 15, 1980, the State and St. Louis Park were granted leave to intervene in the RCRA Section 7003 claim and to assert additional claims under Minnesota law. On or about June 16, 1981, Hopkins was granted leave to intervene in the RCRA Section 7003 claim and to assert additional claims under Minnesota law.

8. On or about September 9, 1981, the United States filed an amended complaint, alleging in addition to the RCRA § 7003 claim, claims under Sections 106 and 107 of the

Comprehensive Environmental Response, Compensation and Liability Act ("CERCLA"), 42 U.S.C. §§ 9606 and 9607.

9. On or about May 27, 1981, the State filed an amended complaint, asserting claims under Section 7003 of RCRA, 42 U.S.C. § 6973, Section 107 of CERCLA, 42 U.S.C. § 9607, Minn. Stat. §§ 115.061, 115.07, 115.071, and Minnesota Rule WPC 4(b) [Minn. Rule Part 7100.0020], and Minnesota common law.

10. On or about August 31, 1981, and October 16, 1981, respectively, St. Louis Park and Hopkins filed amended complaints alleging, inter alia, claims under Section 7003 of RCRA, 42 U.S.C. § 6973, Section 107 of CERCLA, 42 U.S.C. § 9607, Minn. Stat. Chapter 116B, and Minnesota common law.

11. On or about April 5, 1985, the Court granted the State's motion for leave to file a second amended complaint, adding claims under the Minnesota Environmental Response and Liability Act ("MERLA"), Minn. Stat. Ch. 115B. The State subsequently filed such a second amended complaint. Pursuant to stipulations, St. Louis Park and Hopkins later also filed second amended complaints, each of which added MERLA claims.

12. Reilly, in its answers to the various complaints referenced above, has denied and continues to deny liability, has raised several affirmative defenses, and has asserted a counterclaim against St. Louis Park. Various other Parties have asserted cross-claims, including a cross-claim by St. Louis Park against the State, a cross-claim of Oak Park Village

Associates against the Housing and Redevelopment Authority of St. Louis Park and a cross-claim of Philip's Investment Co. against Reilly.

13. Since 1969, a number of studies and/or reports, chemical analyses and field investigations relating to the Site have been undertaken. By listing the items below, the Parties do not necessarily endorse the accuracy, correctness, precision, quality, or validity of the information and opinions contained therein. These analyses, investigations and studies include but are not limited to the following:

(a) Studies and/or Reports

- (1) "Ground Water Investigation Program at St. Louis Park, MN," by E. A. Hickok & Associates, Inc., September, 1969.
- (2) "Memorandum of Waste Disposal at Republic Creosote Co. and Reilly Tar & Chemical Co.," by Minnesota Pollution Control Agency (MPCA Board Item), April, 22, 1970.
- (3) "An Assemblage of Analytical Data Regarding the Reilly Tar & Chemical Property, St. Louis Park, Minnesota," by the St. Louis Park Health Department, August 1, 1972.
- (4) "Status Report on Creosote Site and TexaTonka Area", prepared by the St. Louis Park Planning Department, January 11, 1973.
- (5) "Surface and Subsurface Ground Reclamation; Republic Creosote Site, City of St. Louis Park", prepared by OSM Consulting Engineers, April 23, 1973.

- (6) "Storm Water Study; Public Improvement #72--43 (Republic Creosote Area)," prepared by OSM Consulting Engineers, August 6, 1973.
- (7) "Geology of the St. Louis Park Area - A Review by the Minnesota Geological Survey; Report on Investigation of Municipal Water Supply, St. Louis Park," prepared by the Minnesota Department of Health, March 1974.
- (8) "Soil Investigation; Proposed Storm Sewer and Holding Ponds near Highway 7 and Louisiana Avenue, St. Louis Park," prepared by Soil Exploration Co., April 16, 1974.
- (9) "Hydrogeologic Study of the Republic Creosote Site," prepared by Gerald Sunde, Consulting Engineer, July, 1974.
- (10) "Report on Investigation of Phenol Problem in Private and Municipal Wells in St. Louis Park, Minnesota," prepared by Minnesota Department of Health, September, 1974.
- (11) Memorandum from F. F. Heisel, Minnesota Department of Health, to P. Gove, Minnesota Pollution Control Agency. "St. Louis Park Creosote Contamination Study," November 14, 1975.
- (12) "Data Regarding The History and Development of a Storm Sewer System for the City in the Area of the Former Republic Creosote Property," prepared by the City of St. Louis Park, November 15, 1974.
- (13) "Memorandum on Groundwater Contamination, St. Louis Park, MN," by Minnesota Pollution Control Agency, (MPCA Board Item) November 19, 1974.
- (14) "Memorandum on St. Louis Park Groundwater Situation," by the Minnesota Pollution Control Agency, (MPCA Board Item) December 13, 1974.

- (15) "Soil and Ground Water Investigation Coal Tar Distillation and Wood Preserving Site, St. Louis Park - Phase I Report," prepared by Barr Engineering Co., May 1976.
- (16) "Stability Study of Para Benzo Quinone for the City of St. Louis Park," prepared by Sanitary Engineering Laboratories Inc. (SERCO), June 1976.
- (17) "Soil Boring and Chemical Analysis of the Northern Portion of Oak Park Village," prepared by National Biocentric, Inc., September 17, 1976.
- (18) "Soil Contamination by Creosote Wastes," prepared by National Biocentric, Inc., November 1, 1976.
- (19) "Development Plan, Northern Portion, Oak Park Village," prepared by St. Louis Park, December 2, 1976.
- (20) "Review of Recent Studies of Soil Contamination at the Former Republic Creosote Site - Recommendations to City's Proposed Development Plan," by Minnesota Pollution Control Agency, December 28, 1976.
- (21) "Soil and Ground Water Investigation Coal Tar Distillation and Wood Preserving Site, St. Louis Park - Phase II Report," prepared by Barr Engineering Co., June 1977.
- (22) "Assessment of Possible Human Health Effects Resulting from Contamination of the Former Republic Creosote Site," prepared by the Minnesota Department of Health, October 1977.
- (23) "Soil Report; Prepared by Oak Park Village, St. Louis Park, Minnesota," prepared by Soil Testing Service of Minnesota, Inc., January 5, 1978.
- (24) "Recommendations for Plugging or Modification of Abandoned Wells in the

Area of the Former Republic Creosote Plant," prepared by the City of St. Louis Park, January 11, 1978.

- (25) "Report of Well Water Survey, St. Louis Park, Minnesota," prepared by Sanitary Engineering Laboratories, Inc., (SERCO) June-July 1978.
- (26) "Report on the Existing Creosote Problem in St. Louis Park, Minnesota," prepared by James Bailey, Agricultural Engineering, University of Minnesota, July 1, 1978.
- (27) "Health Implications of Polynuclear Aromatic Hydrocarbons in St. Louis Park Drinking Water," prepared by the Minnesota Department of Health, November 1978.
- (28) "Status Report to the MPCA: Proposed Development, Oak Park Village," prepared by St. Louis Park, November 14, 1978.
- (29) "Water Quality Development in Oak Park Village," prepared by St. Louis Park Planning Department, December 15, 1978.
- (30) "Letter Report Tabulating Information on Existing Wells in St. Louis Park," prepared by United States Geological Survey, February 6, 1979.
- (31) "Status Report: St. Louis Park Development," by the Minnesota Pollution Control Agency (MPCA Board Item), March 27, 1979.
- (32) "Progress Report: Investigation of Coal Tar Derivatives in Ground Water - St. Louis Park," prepared by the United States Geological Survey, April 13, 1979.
- (33) "Epidemiologic Investigation of Third National Cancer Survey Data for St. Louis Park, Edina, Richfield and Minneapolis St. Paul SMSA with a

Historical Review of St. Louis Park's Water Supply," prepared by Kari Dusich, September 1979.

- (34) "Emergency Pumpout Well For Reilly Tar Site, St. Louis Park, Minnesota," prepared by Ecology and Environment, Inc., 1980.
- (35) "Examination of Cost Estimate For Three Tasks to be Completed For The Reilly Tar and Chemical Project, St. Louis Park, MN," prepared by Ecology and Environment, Inc., 1980.
- (36) "Summary Report on the City of St. Louis Park Activated Carbon Pilot Plant Study," prepared by Sanitary Engineering Laboratories, Inc., (SERCO), January 11, 1980.
- (37) "Cancer Rates in a Community Exposed to Low Levels of Creosote Components in Municipal Water," prepared by Dusich, Sigurdson, Hall, Dean, Minnesota Medicine, November 1980.
- (38) "Preliminary Evaluation of Ground Water Contamination by Coal Tar Derivative, St. Louis Park, MN," prepared by the United States Geological Survey, January 1981.
- (39) "Report on Drinking Water Treatment and Remedy Evaluation for St. Louis Park, MN," prepared by Eugene A. Hickok and Associates, Inc., April 1981.
- (40) "Report and Statistic - Water Quality: Results of St. Louis Park Water Samples," prepared by H. Taylor, United States Geological Survey, June 10, 1981.
- (41) "Study of Ground Water Contamination in St. Louis Park, MN," prepared by Eugene A. Hickok & Associates, et. al., November 1981.
- (42) "Dispersion and Sorption of Hydrocarbons in Aquifer Material," by

G. Cohn (thesis) University of Minnesota, 1982.

- (43) "Terminating An Endless Search: An Action Approach to Solving the Water Problem," prepared by St. Louis Park, January 11, 1982.
- (44) "Request for Authorization to Negotiate and Enter into Cooperative Agreement with the U.S. EPA to Obtain Funds for Additional Cleanup Work at the Reilly Tar Site, St. Louis Park," by the Minnesota Pollution Control Agency (MPCA Board Item), May 25, 1982.
- (45) "Degradation of Phenolic Contaminants in Ground Water by Anaerobic Bacteria: St. Louis Park, MN," prepared by Erlich, Goerlitz, Godsy & Hult, United States Geological Survey, November 1982.
- (46) "Evaluation of Groundwater Treatment and Water Supply Alternatives for St. Louis Park, MN," prepared by CH2M Hill, 1982-1983.
- (47) "Recommended Plan for a Comprehensive Solution of the Polynuclear Aromatic Hydrocarbon Contamination Problem in the St. Louis Park Area," prepared by Environmental Research & Technology, Inc. for Reilly Tar & Chemical Corporation, April 1983, plus Errata, June 27, 1983 and November 27, 1984.
- (48) "Health Risk Assessment and Environmental Effects of Compounds Contaminating St. Louis Park Groundwater: Selected Two - and Three - Ring Heterocycles and Indene," prepared by Stephen M. Mabley, Minnesota Department of Health, Section of Health Risk Assessment, July 1983.
- (49) "Evaluation of Activated Carbon Treatment Alternative for Polynuclear Aromatic Hydrocarbon Removal for Groundwater in the St. Louis Park Area," prepared by Calgon Carbon Corporation, November 18, 1983.

- (50) "Request for Authorization to Negotiate and Execute an Amendment to the Current Cooperative Agreement with the U.S. Environmental Protection Agency for Investigation and Remedial Action at the Reilly Tar and Chemical Company Hazardous Waste Site in St. Louis Park," by the Minnesota Pollution Control Agency (MPCA Board Item), November 22, 1983.
- (51) "Assessment of Groundwater Contamination by Coal Tar Derivatives, St. Louis Park Area, MN", prepared by M. F. Hult, United States Geological Survey, Open File Report 64-867, 1984.
- (52) "Record of Decision, Remedial Action Alternative Selection," prepared by the United States Environmental Protection Agency, June 6, 1984.
- (53) "Evaluation of Granular Activated Carbon for the Removal of Polynuclear Aromatic Hydrocarbons from Municipal Well Water in St. Louis Park, MN," prepared by Calgon Carbon Corporation, September 10, 1984.
- (54) "Sampling and Analysis Plan for Calgon Accelerated Column Testing of SLP 15 Water," prepared by Environmental Research & Technology, Inc., October 25, 1984.
- (55) "Request for Issuance of a Request for Response Action to the Reilly Tar and Chemical Corporation Regarding Contamination At and Around the Reilly Tar Hazardous Waste Site in St. Louis Park," by the Minnesota Pollution Control Agency (MPCA Board Item), December 18, 1984.
- (56) "Ground-water Flow in Prairie du Chien Jordan Aquifer Related to Contamination by Coal Tar Derivatives, St. Louis Park, MN," prepared by J. R. Stark and M. F. Hult, United States Geological Survey, 1985.

- (57) "Calgon ACT Study: Initial Results from the Accelerated Column Test of PAH Removal Performance for Activated Carbon Treatment of Water From SLP 15," prepared by Twin City Testing, January 11, 1985.
- (58) "Calgon ACT Study: Further Results From the Study of PAH Removal by Activated Carbon Treatment," prepared by Twin City Testing, January 30, 1985.
- (59) "Keilly Tar and Chemical: Analysis of Water From Three St. Peter Wells," prepared by Twin City Testing, January 31, 1985.
- (60) "Accelerated Column Test for Removal of Polynuclear Aromatic Hydrocarbons from Contaminated Groundwater," prepared by Calgon Corporation, March 8, 1985.
- (61) "PAH Analysis by GCMS," prepared by Twin City Testing March 26, 1985
- (62) "Draft Work Plan R1, Reilly Tar Site, St. Louis Park, Minnesota," prepared by CH2M Hill and Ecology & Environment, April 27, 1985.
- (63) "Predesign Memorandum Evaluation of Granular Activated Carbon System Alternatives For Removal of Polynuclear Aromatic Hydrocarbons From Municipal Well Water in St. Louis Park, Minnesota", prepared by Ch2M Hill, May 29, 1985.
- (64) "PAH Threshold Odor Determination in St. Louis Park Municipal Supply Water," prepared by Environmental Research and Technology, Inc., May 30, 1985.
- (65) "Volatile Organic Analysis of the St. Louis Park Municipal Drinking Water Supply System, March, 1985," prepared by Environmental Research & Technology, Inc., May 30, 1985.
- (66) Feasibility of Community-Wide Epidemiologic Studies of Drinking Water and Health: St. Louis Park and New Brighton", prepared by the Minnesota Department of Health, December 31, 1985.

(b) Field investigations and chemical analyses of water (surface and/or ground water) and soils, including associated field notes, chain of custody records, raw data sheets, sampling analysis protocols, boring and well logs and water level measurements. In general, the results of soil borings and water samples are found in the list of studies and/or reports under Part C. 13(a). (Dates listed usually reflect the time of the investigation.)

- (1) Preliminary soil investigation for the engineering properties of the soil, performed by Soil Engineering Services, Inc., October 13, 1969.**
- (2) Mellon-Rice data on well water and plant wastewater samples, Carnegie-Mellon University and C.W. Rice Division, NUS, November 5, 1970.**
- (3) Soil sample analyses, Tri-City Public Health Lab, 1971 and 1973.**
- (4) Analysis of soil and water samples from the St. Louis Park area, by the Minnesota Department of Health, 1973 to present.**
- (5) Analysis of soil and water samples by Twin Cities Testing and Engineering Laboratory, Inc., and Soil Exploration Company, 1974 to present.**
- (6) Analysis of soil and water samples by Sanitary Engineering Laboratories, Inc. (SERCO), 1975, 1976, 1977, 1978 and 1982.**
- (7) Soil borings performed by Braun Engineering, 1974, 1979, 1980, and 1982.**
- (8) Well investigations pursuant to well abandonment program performed by Minnesota Department of Health, 1978-present.**
- (9) Analysis of soil and water by United States Geological Survey, 1978-present.**

- (10) Analyses of groundwater, by Pace Laboratories, Inc., 1978-1980, 1983-1984 (1983-1984 analyses performed by Rocky Mountain Analytical Laboratory).
- (11) "Results of Analysis of Water Samples, and Soil Samples for Polynuclear Aromatic Compounds (Hydrocarbons, Azarene, Phenols)", by Midwest Research Institute, October 7, 1981.
- (12) Analyses of Ground Water, by Capsule Laboratories, Inc., 1981, 1982, and 1983.
- (13) Soil borings and analyses by GCA Corp., 1982-1983.
- (14) Water analyses by Monsanto Research Corp., 1982-1984.
- (15) Water analyses by Environmental Testing and Certification Corporation, 1983.
- (16) Soil boring and chemical analyses by National Biocentric, Inc., 1976.
- (17) St. Louis Park area water well search and inventory questionnaires, prepared by E. A. Hickok and Associates, Inc., 1982-1983.
- (18) Progress reports on the investigation and clean-out of W23 and W105, E.A. Hickok & Associates, Inc., 1982 to present.
- (19) Water samples and analyses by CH2M Hill, 1982 and 1983.
- (20) Water samples and analyses by Environmental Research and Technology, Inc., 1982 to present.
- (21) Water samples and analyses by Acurex Corporation, 1984 to present.
- (22) Water analyses by United States Environmental Protection Agency 1977 and 1981-1982.

HEALTH AND SAFETY PLAN

HEALTH AND SAFETY PLAN

Introduction

This Health and Safety Plan applies to personnel who will potentially be exposed to groundwater affected by creosote or coal tar constituents during the retrieval of groundwater samples from active pumping wells, the GAC plant, monitor wells, and piezometers. This plan has been designated to comply with, as a minimum, the requirements set forth in 29 CFR 1910.120, the OSHA standards governing hazardous waste operations. In no case may work be performed in a manner that conflicts with the intent of or the safety concerns expressed in this plan.

Materials of Concern and effects of Overexposure

The materials of concern which have been identified for this project are coal tar and creosote related materials including naphthalene, other polynuclear aromatic hydrocarbons (PAH) and phenolic compounds.

Coal tar and creosote are typically irritating to the eyes, skin and respiratory tract. Acute skin contact may cause burning and itching while prolonged contact and poor hygiene practices may produce dermatitis. Prolonged skin contact with creosote must be avoided to prevent the possibility of skin absorption.

Naphthalene is a hemolytic agent which, upon overexposure to the vapor or ingestion of the solid, may produce a variety of symptoms associated with the breakdown of red blood cells. Naphthalene is also irritating to the eyes and repeated or prolonged contact has been associated with the production of cataracts.

Repeated exposure to certain PAH compounds has been associated with the production of cancer. Contact of PAH compounds with the skin may cause photosensitization of the skin producing skin burns after subsequent exposure to ultraviolet radiation.

Phenolics are generally strong irritants which can have a corrosive effect on the skin and can also rapidly penetrate the skin. Overexposure to phenols and phenolic compounds may cause convulsions as well as liver and kidney damage.

Hazard Assessment

Initial

Because of the relatively low vapor pressures associated with PAH compounds (generally less than 10^{-4} mm Hg at 20°C), they are not expected to present a vapor hazard. The most likely threat of exposure to these compounds will be via skin contact.

TABLE 1
ACTION LIMITS FOR AIR CONTAMINANTS

<u>Limit</u>	<u>Persistent Concentration in the Breathing Zone</u>	<u>Procedure</u>
Lower	5 ppm	Don respirators, step up monitoring.
Upper	50 ppm	Stop work and back off from immediate work area until levels subside in the breathing zone.

Action Limits

The American Conference of Governmental Industrial Hygienists (ACGIH) has established threshold limit values (TLV) for phenol and naphthalene at 5 and 10 ppm, respectively, as 8-hour time weighted averages (TWA). Based on these values, the action limits in Table 1 have been set. The lower limit of 5 ppm is based on the TLV for phenol while the upper limit of 50 ppm is based on a minimum protection factor of 10 for a half-mask, air purifying respirator.

Response

When the PID yields persistent breathing-zone readings at or above the lower action limit, workers in the affected area will don respirators. Air sampling will continue on a more frequent basis. If readings are persistent at or above the upper limit, workers shall back off from the immediate work area until measured breathing-zone concentrations fall below the lower limit, at which time operations will resume and normal air monitoring will continue. If breathing zone levels do not fall below the upper limit, workers are to leave the work area and report the condition immediately to the City, the Engineer, or its representative. If necessary, engineering controls will be instituted to maintain vapor concentrations below the upper limit or arrangements will be made to upgrade to Level B protection.

Personal Protective Equipment

Personal protective equipment (PPE) will be donned, as necessary, based on the hazards encountered. Listed below is the personal protective equipment to be utilized during this project and the conditions requiring its use.

Personal Protective Equipment

- Coveralls - Polyethylene coated Tyvek if work involves contact with affected soil or groundwater.
- Boots - Chemical resistant type if work involves contact with affected soil or groundwater.
- Hard Hat - When working in the vicinity of operating heavy machinery.
- Face shield - If splash hazard exists.
- Gloves - Nitrile for potential contact with affected soil or groundwater.
- Respirator - MSA Comfo II with GMC-H Cartridges if PID reading exceeds 5 ppm or if dust or odors become objectionable.
- Chemical Safety Goggles - If eye irritation occurs.

Because of the carcinogenicity of certain PAH compounds, and because of the skin hazards associated with PAH and phenolic compounds, it is important that appropriate protective clothing be worn during work activities, which may involve the possibility of skin contact with affected soil or groundwater. As a minimum, the presence of visible creosote or coal tar related material shall constitute evidence of affected soil or groundwater.

Health and Safety Training

Personnel covered by this Health and Safety Plan must have received appropriate health and safety training prior to their working on the site. Training will include:

- Requirements for and use of respirators and personal protective equipment.
- Required personal hygiene practices.
- Requirements for employees to work in pairs.
- Proper material handling.
- Proper sampling procedures.
- Maintenance of safety equipment.
- Effective response to any emergency.
- Emergency procedures.
- Hazard zones.
- Decontamination methods.
- General safety precautions.

A copy of the Standard Safety Procedures (Table 2) will be given to each worker covered by this Health and Safety Plan.

Decontamination

Administrative procedures require hygienic practices consistent with work hazards. Employees will be instructed in the training program on proper personal hygiene procedures.

Contaminated, reuseable PPE, such as boots, hard hats, face shields and goggles, will be decontaminated prior to leaving the site. The decontamination procedure follows:

- Rinse with water to remove gross contamination.
- Wash in Alconox or equivalent detergent solution.
- Rinse with clean water.

Contaminated, disposable PPE, such as Tyvek coveralls and gloves will be placed in 55-gallon drums and stored while arrangements are made for disposal.

TABLE 2
STANDARD SAFETY PROCEDURES

- Employees are required to work in pairs.
- Wash face and hands prior to eating, smoking, or leaving the site.
- No smoking or eating is allowed in the work area during excavation or sampling activities.
- Wearing of contact lenses is not permitted in the work area.
- Contaminated material (e.g., Tyvek coveralls) must be properly disposed of before leaving the site.
- All work must be conducted in accordance with local, state and federal EPA and OSHA regulations, particularly 29 CFR 1910.120.

Respirators, if used, will be cleaned and disinfected after each day of use. The facepiece (with cartridge removed) will be washed in a hypochlorite (or equivalent) disinfecting solution, rinsed in warm water and air dried in a clean place.

Emergency Procedures

This Health and Safety Plan has been established to allow site operations to be conducted without adverse impacts on worker health and safety as well as public health and safety. In addition, supplementary emergency response procedures have been developed to cover extraordinary conditions at the site.

General

All accidents and unusual events will be dealt with in a manner to minimize a continued health risk to site workers. In the event that an accident or other unusual event occurs, the following procedure will be followed:

- First aid or other appropriate initial action will be administered by those closest to the accident/event. This assistance will be conducted so that those rendering assistance are not placed in a situation of unacceptable risk. In the event that a worker is caught in a trench collapse, call for emergency assistance immediately.
- All accidents/unusual events must be immediately reported to the Owner.
- All workers on site should conduct themselves in a mature, calm manner in the event of an accident/unusual event, to avoid spreading the danger to themselves, surrounding workers and the community.

Responses to Specific Situations

Emergency procedures for specific situations are given in the following paragraphs.

Worker Injury

If an employee in an affected area is physically injured, Red Cross first-aid procedures will be followed. Depending on the severity of the injury, emergency medical response may be sought.

If the injury to the worker is chemical in nature (e.g., overexposure), the following first-aid procedures are to be instituted:

- Eye Exposure - If affected solids or liquids get into the eyes, wash eyes immediately using large amounts of water and lifting the lower and upper lid occasionally. Obtain medical attention immediately.
- Skin Exposure - If affected solids or liquids get on the skin, promptly wash the affected skin using soap or mild detergent and water. Obtain medical attention immediately when exposed to concentrated solids or liquids.

- Inhalation - If a person inhales large amounts of a toxic vapor, move the exposed person to fresh air at once. If breathing has stopped, perform artificial respiration. Keep the affected person warm and at rest. Obtain medical attention as soon as possible.
- Swallowing - When affected solids or liquids have been swallowed, the Poison Control Center will be contacted and their recommended procedures followed.

Emergency Notification

In an extraordinary event that might be damaging to personnel or adjacent property, immediate notification of the proper emergency service will be required. The proper emergency service is determined by the nature of the emergency.

EMERGENCY NOTIFICATION

Fire Department	911
Ambulance	911
Police Department	911
Methodist Hospital	932-5000
Poison Control Center	347-3141

OTHER CONTACTS

MPCA - Michael Vennewitz	612-296-7782
EPA - Erin Moran	312-886-7238
City of St. Louis Park - James Grube .	612-924-2551
- William Gregg (ERT, Inc.)	612-924-0117

SECTION D
COMMUNITY RELATIONS PLAN

COMMUNITY RELATIONS PLAN

The Initial Sampling Plan is to be completed in accordance with the Consent Decree - Remedial Action Plan for Reilly Tar & Chemical Corporation's St. Louis Park, Minnesota, N.P.L. Site. All community relations programs related to this work will be coordinated through the following agencies:

United States

Ms. Judy Beck

**United States Environmental Protection Agency
(312) 353-1325**

State of Minnesota

Ms. Susan Brustman

**Minnesota Pollution Control Agency
(612) 296-7769**

City of St. Louis Park

Ms. Sharon Klumpp

**City of St. Louis Park
(612) 924-2523**

Information necessary to conduct the Community Relations Plan will be provided by the City and Reilly.